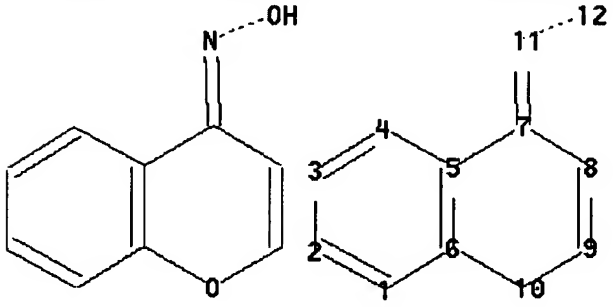
HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):0

=>
Uploading C:\Program Files\Stnexp\Queries\10959610A.str



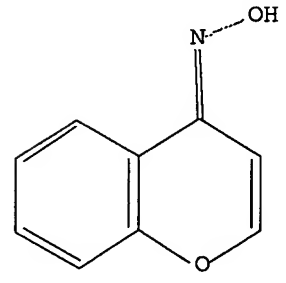
chain nodes :
11 12
ring nodes :
1 2 3 4 5 6 7 8 9 10
chain bonds :
7-11 11-12
ring bonds :
1-2 1-6 2-3 3-4 4-5 5-6 5-7 6-10 7-8 8-9 9-10
exact/norm bonds :
5-7 6-10 7-8 7-11 8-9 9-10 11-12
normalized bonds :
1-2 1-6 2-3 3-4 4-5 5-6

G1

Match level :
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:Atom 8:Atom 9:Atom 10:Atom
11:CLASS 12:CLASS

L3 STRUCTURE UPLOADED

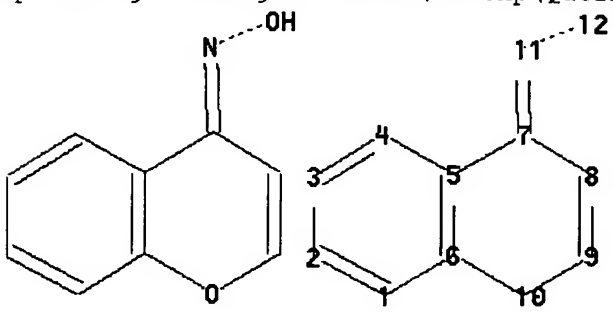
=> d l3
L3 HAS NO ANSWERS
L3 STR



G1

Structure attributes must be viewed using STN Express query preparation.

=>
Uploading C:\Program Files\Stnexp\Queries\10808678.str

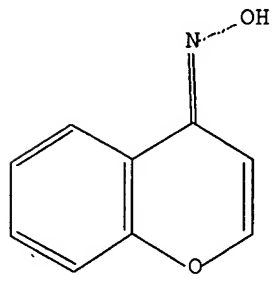


chain nodes :
11 12
ring nodes :
1 2 3 4 5 6 7 8 9 10
chain bonds :
7-11 11-12
ring bonds :
1-2 1-6 2-3 3-4 4-5 5-6 5-7 6-10 7-8 8-9 9-10
exact/norm bonds :
5-7 6-10 7-8 7-11 8-9 9-10 11-12
normalized bonds :
1-2 1-6 2-3 3-4 4-5 5-6

Match level :
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:Atom 8:Atom 9:Atom 10:Atom
11:CLASS 12:CLASS

L4 STRUCTURE UPLOADED

=> d l4
L4 HAS NO ANSWERS
L4 STR



Structure attributes must be viewed using STN Express query preparation.

=> s sss l4 sam
SAMPLE SEARCH INITIATED 18:00:58 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED - 1141 TO ITERATE

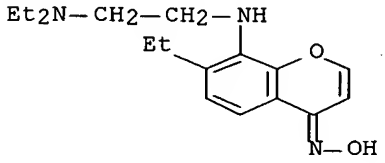
100.0% PROCESSED 1141 ITERATIONS 12 ANSWERS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED ITERATIONS: 20794 TO 24846
PROJECTED ANSWERS: 33 TO 447

L5 12 SEA SSS SAM L4

=> d scan

L5 12 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Chromone, 8-[(2-diethylaminoethyl)amino]-7-ethyl-, oxime (6CI)
MF C17 H25 N3 O2



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):0

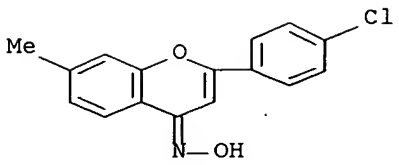
=> s sss l4 full
FULL SEARCH INITIATED 18:01:31 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 22736 TO ITERATE

100.0% PROCESSED 22736 ITERATIONS 276 ANSWERS
SEARCH TIME: 00.00.01

L6 276 SEA SSS FUL L4

=> d scan

L6 276 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-chlorophenyl)-7-methyl-, oxime (9CI)
MF C16 H12 Cl N O2

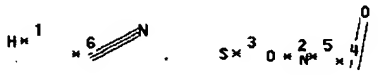


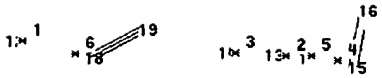
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

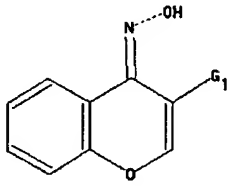
HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):0

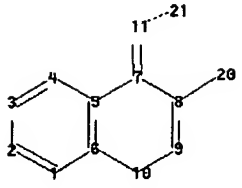
=>
=> save l6 gree108678/A
ANSWER SET L6 HAS BEEN SAVED AS 'GREE108678/A'

=>
Uploading C:\Program Files\Stnexp\Queries\10808678A.str









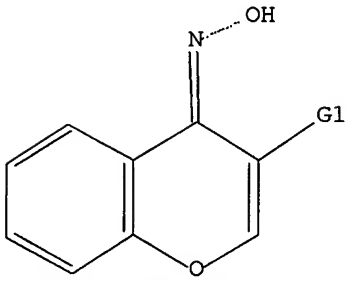
chain nodes :
11 12 13 14 15 16 17 18 19 20 21
ring nodes :
1 2 3 4 5 6 7 8 9 10
chain bonds :
7-11 8-20 11-21 15-16 18-19
ring bonds :
1-2 1-6 2-3 3-4 4-5 5-6 5-7 6-10 7-8 8-9 9-10
exact/norm bonds :
5-7 6-10 7-8 7-11 8-9 8-20 9-10 11-21 15-16 18-19
normalized bonds :
1-2 1-6 2-3 3-4 4-5 5-6

G1:[*1],[*2],[*3],[*4],[*5],[*6]

Match level :
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:Atom 8:Atom 9:Atom 10:Atom
11:CLASS 12:CLASS 13:CLASS 14:CLASS 15:CLASS 16:CLASS 17:CLASS 18:CLASS
19:CLASS 20:CLASS
21:CLASS

L7 STRUCTURE UPLOADED

=> d l7
L7 HAS NO ANSWERS
L7 STR



G1 [@1],[@2],[@3],[@4],[@5],[@6]

Structure attributes must be viewed using STN Express query preparation.

=> s sss l7 subset=l6 full
FULL SUBSET SEARCH INITIATED 18:04:56 FILE 'REGISTRY'
FULL SUBSET SCREEN SEARCH COMPLETED - 276 TO ITERATE

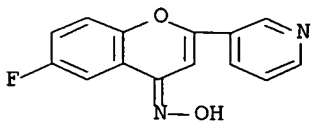
100.0% PROCESSED 276 ITERATIONS 230 ANSWERS
SEARCH TIME: 00.00.01

L8 230 SEA SUB=L6 SSS FUL L7

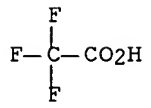
=> d scan

L8 230 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 6-fluoro-2-(3-pyridinyl)-, oxime, mono(trifluoroacetate) (9CI)
MF C14 H9 F N2 O2 . C2 H F3 O2

CM 1



CM 2



HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):0

=> fil caplus
COST IN U.S. DOLLARS

	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	219.50	219.71

FILE 'CAPLUS' ENTERED AT 18:05:21 ON 11 JUL 2007
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

FILE COVERS 1907 - 11 Jul 2007 VOL 147 ISS 3
FILE LAST UPDATED: 10 Jul 2007 (20070710/ED)

Effective October 17, 2005, revised CAS Information Use Policies apply. They are available for your review at:

http://www.cas.org/infopolicy.html

=> s l8
L9 66 L8

=> s l9 and (ay<2003 or py<2003 or pry<2003)
4447904 AY<2003
22885817 PY<2003
3926313 PRY<2003
L10 64 L9 AND (AY<2003 OR PY<2003 OR PRY<2003)

=> d scan

L10 64 ANSWERS CAPLUS COPYRIGHT 2007 ACS on STN
CC 37 (Heterocyclic Compounds (One Hetero Atom))
TI Linear benzochromanones and benzochromones. II. Synthesis of 6,7-cyclohexenochromanone, 6,7-benzochromanone, 6,7-cyclohexenochromone, and 6,7-benzochromone

IT Spectra, visible and ultraviolet
(of 4H-naphtho[2,3-b]pyran-4-one derivs.)
IT 17056-93-8 40420-05-1
(Derived from data in the 7th Collective Formula Index (1962-1966))
IT 1125-78-6P, 2-Naphthol, 5,6,7,8-tetrahydro- 4707-29-3P, 4H-Naphtho[2,3-b]pyran-4-one, 6,7,8,9-tetrahydro- 4707-38-4P, 4H-Naphtho[2,3-b]pyran-4-one 4707-39-5P, 4H-Naphtho[2,3-b]pyran-4-one, 2,3-dihydro- 4707-40-8P, 4H-Naphtho[2,3-b]pyran-4-one, 2,3,6,7,8,9-hexahydro- 89228-44-4P, 2-Naphthol, 5,6,7,8-tetrahydro-, acetate 91909-81-8P, Acetaldehyde, (5,6,7,8-tetrahydro-3-hydroxy-2-naphthoyl)- 93187-69-0P, Propionitrile, 3-[(5,6,7,8-tetrahydro-2-naphthyl)oxy]- 93944-33-3P, Acetaldehyde, (3-hydroxy-2-naphthoyl)- 96200-32-7P, 4H-Naphtho[2,3-b]pyran-4-one, oxime 96447-08-4P, 4H-Naphtho[2,3-b]pyran-4-one, 2,3,6,7,8,9-hexahydro-, oxime 96679-42-4P, 4H-Naphtho[2,3-b]pyran-4-one, 6,7,8,9-tetrahydro-, oxime 97024-60-7P, Propionic acid, 3-[(5,6,7,8-tetrahydro-2-naphthyl)oxy]- 98655-54-0P, 4H-Naphtho[2,3-b]pyran-4-one, 2,3-dihydro-, (2,4-dinitrophenyl)hydrazone 98783-40-5P, 4H-Naphtho[2,3-b]pyran-4-one, 2,3,6,7,8,9-hexahydro-, (2,4-dinitrophenyl)hydrazone 875218-34-1P, 2'-Acetonaphthone-6'-(acetonyloxy)-, 3'-hydroxy- 875834-53-0P, 2'-Acetonaphthone-6'-(acetonyloxy)-, 5',6',7',8'-tetrahydro-3'-hydroxy-
RL: PREP (Preparation)
(preparation of)

HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):2

L10 64 ANSWERS CAPLUS COPYRIGHT 2007 ACS on STN
CC 10 (Organic Chemistry)
TI Hydroxycarbonyl compounds. X. Coumarins and chromones from m-4-xylenol
IT Carbonyl compounds
(hydroxy)
IT Chromone, 2-ethyl-6,8-dimethyl-3-propionyl-, oxime
RL: PREP (Preparation)
IT 105-67-9, 2,4-Xylenol
(and derivs.)
IT 91-64-5, Coumarin 491-38-3, Chromone
(derivs.)
IT 1076-97-7P, 1,4-Cyclohexanedicarboxylic acid 5570-72-9P, Propiophenone, 2-hydroxy-3,5-dimethyl- 51233-78-4P, Propionic acid, 2,4-xylyl ester 84437-35-4P, Chromone, 2,6,8-trimethyl- 84437-35-4P, Chromone, 2,6,8-trimethyl- 89228-74-0P, Coumarin, 4,6,8-trimethyl- 104516-36-1P, 1,3-Butanedione, 1-(2-hydroxy-3,5-xylyl)- 123491-20-3P, Chromone, 2,3,6,8-tetramethyl- 717916-12-6P, Acetophenone, 2-methoxy-3,5-dimethyl-, semicarbazone 723755-49-5P, Butyric acid, 2,4-xylyl ester 853996-48-2P, Chromone, 3-ethyl-2,6,8-trimethyl- 857783-25-6P, Coumarin, 3-ethyl-4,6,8-trimethyl- 857824-12-5P, Flavone, 3-benzoyl-6,8-dimethyl- 859085-50-0P, Chromone, 2-ethyl-6,8-dimethyl- 859085-93-1P, Chromone, 3,6,8-trimethyl-2-(3,4-methylenedioxystyryl)- 859175-36-3P, Cinnamic acid, α-ethyl-2-methoxy-β,3,5-trimethyl- 859176-56-0P, Cinnamic acid, 2-methoxy-β,3,5-trimethyl- 859176-64-0P, Cinnamic acid, 2-methoxy-α,β,3,5-tetramethyl- 859805-44-0P, Chromone, 3-ethyl-6,8-dimethyl-2-(3,4-methylenedioxystyryl)- 859805-59-7P, Chromone, 6,8-dimethyl-2-(3,4-methylenedioxystyryl)- 859805-98-4P, Chromone, 3-acetyl-2,6,8-trimethyl-, oxime 859821-52-6P, Coumarin, 6,8-dimethyl-4-phenyl- 859821-95-7P, Coumarin, 3-benzyl-4,6,8-trimethyl- 873989-36-7P, Butyrophenone (PhCOCH2CH2CH3), 2-hydroxy-3,5-dimethyl-
RL: PREP (Preparation)
(preparation of)

```
L10   64 ANSWERS   CAPLUS  COPYRIGHT 2007 ACS on STN
CC    37 (Heterocyclic Compounds (One Hetero Atom))
TI    Polyfluoro derivatives of γ-benzopyrone
IT    4487-62-1P, 4H-1-Benzopyran-3-carboxylic acid,
      5,6,7,8-tetrafluoro-2-methyl-4-oxo-, ethyl ester, oxime   209736-41-4P,
      4H-1-Benzopyran-3-carboxylic acid, 5,6,7,8-tetrafluoro-2-methyl-4-oxo-
      RL: PREP (Preparation)
         (preparation of)

HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):0

=> s l9 and kinase?inhibition
'?' TRUNCATION SYMBOL NOT VALID WITHIN 'KINASE?INHIBITION'
The truncation symbol ?  may be used only at the end of a search
term.  To specify a variable character within a word use '!', e.g.,
'wom!n' to search for both 'woman' and 'women'.  Enter "HELP
TRUNCATION" at an arrow prompt (=>) for more information.

=> s l9 and ?kinase?inhibition
'?' TRUNCATION SYMBOL NOT VALID WITHIN '?KINASE?INHIBITION'
The truncation symbol ?  may be used only at the end of a search
term.  To specify a variable character within a word use '!', e.g.,
'wom!n' to search for both 'woman' and 'women'.  Enter "HELP
TRUNCATION" at an arrow prompt (=>) for more information.

=> s l9 and ?kinase?
        344183 ?KINASE?
L11          1 L9 AND ?KINASE?

=> d his

     (FILE 'HOME' ENTERED AT 17:55:47 ON 11 JUL 2007)

     FILE 'REGISTRY' ENTERED AT 17:56:03 ON 11 JUL 2007
L1               STRUCTURE UPLOADED
L2             9 S SSS L1 SAM
L3               STRUCTURE UPLOADED
L4               STRUCTURE UPLOADED
L5            12 S SSS L4 SAM
L6           276 S SSS L4 FULL
                 SAVE L6 GREE108678/A
L7               STRUCTURE UPLOADED
L8           230 S SSS L7 FULL SUB=L6

     FILE 'CAPLUS' ENTERED AT 18:05:21 ON 11 JUL 2007
L9            66 S L8
L10           64 S L9 AND (AY<2003 OR PY<2003 OR PRY<2003)
L11            1 S L9 AND ?KINASE?

=> d ibib l9 abs hitst 1-66
'HITST' IS NOT A VALID FORMAT FOR FILE 'CAPLUS'

The following are valid formats:

ABS --------- GI and AB
ALL --------- BIB, AB, IND, RE
APPS -------- AI, PRAI
BIB --------- AN, plus Bibliographic Data and PI table (default)
CAN --------- List of CA abstract numbers without answer numbers
CBIB -------- AN, plus Compressed Bibliographic Data
```

```
CLASS ------- IPC, NCL, ECLA, FTERM
DALL -------- ALL, delimited (end of each field identified)
DMAX -------- MAX, delimited for post-processing
FAM --------- AN, PI and PRAI in table, plus Patent Family data
FBIB -------- AN, BIB, plus Patent FAM
IND --------- Indexing data
IPC --------- International Patent Classifications
MAX --------- ALL, plus Patent FAM, RE
PATS -------- PI, SO
SAM --------- CC, SX, TI, ST, IT
SCAN -------- CC, SX, TI, ST, IT (random display, no answer numbers;
              SCAN must be entered on the same line as the DISPLAY,
              e.g., D SCAN or DISPLAY SCAN)
STD --------- BIB, CLASS

IABS -------- ABS, indented with text labels
IALL -------- ALL, indented with text labels
IBIB -------- BIB, indented with text labels
IMAX -------- MAX, indented with text labels
ISTD -------- STD, indented with text labels

OBIB -------- AN, plus Bibliographic Data (original)
OIBIB ------- OBIB, indented with text labels

SBIB -------- BIB, no citations
SIBIB ------- IBIB, no citations

HIT --------- Fields containing hit terms
HITIND ------ IC, ICA, ICI, NCL, CC and index field (ST and IT)
              containing hit terms
HITRN ------- HIT RN and its text modification
HITSTR ------ HIT RN, its text modification, its CA index name, and
              its structure diagram
HITSEQ ------ HIT RN, its text modification, its CA index name, its
              structure diagram, plus NTE and SEQ fields
FHITSTR ----- First HIT RN, its text modification, its CA index name, and
              its structure diagram
FHITSEQ ----- First HIT RN, its text modification, its CA index name, its
              structure diagram, plus NTE and SEQ fields
KWIC -------- Hit term plus 20 words on either side
OCC --------- Number of occurrence of hit term and field in which it occurs

To display a particular field or fields, enter the display field
codes.  For a list of the display field codes, enter HELP DFIELDS at
an arrow prompt (=>).  Examples of formats include: TI; TI,AU; BIB,ST;
TI,IND; TI,SO.  You may specify the format fields in any order and the
information will be displayed in the same order as the format
specification.

All of the formats (except for SAM, SCAN, HIT, HITIND, HITRN, HITSTR,
FHITSTR, HITSEQ, FHITSEQ, KWIC, and OCC) may be used with DISPLAY ACC
to view a specified Accession Number.
ENTER DISPLAY FORMAT (BIB):end

=> d ibib abs hitst l9 1-66
'HITST' IS NOT A VALID FORMAT FOR FILE 'CAPLUS'

The following are valid formats:

ABS --------- GI and AB
```

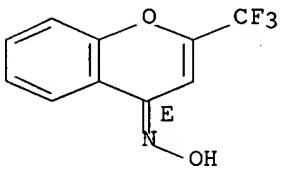
```
ALL --------- BIB, AB, IND, RE
APPS -------- AI, PRAI
BIB --------- AN, plus Bibliographic Data and PI table (default)
CAN --------- List of CA abstract numbers without answer numbers
CBIB -------- AN, plus Compressed Bibliographic Data
CLASS ------- IPC, NCL, ECLA, FTERM
DALL -------- ALL, delimited (end of each field identified)
DMAX -------- MAX, delimited for post-processing
FAM --------- AN, PI and PRAI in table, plus Patent Family data
FBIB -------- AN, BIB, plus Patent FAM
IND --------- Indexing data
IPC --------- International Patent Classifications
MAX --------- ALL, plus Patent FAM, RE
PATS -------- PI, SO
SAM --------- CC, SX, TI, ST, IT
SCAN -------- CC, SX, TI, ST, IT (random display, no answer numbers;
              SCAN must be entered on the same line as the DISPLAY,
              e.g., D SCAN or DISPLAY SCAN)
STD --------- BIB, CLASS

IABS -------- ABS, indented with text labels
IALL -------- ALL, indented with text labels
IBIB -------- BIB, indented with text labels
IMAX -------- MAX, indented with text labels
ISTD -------- STD, indented with text labels

OBIB -------- AN, plus Bibliographic Data (original)
OIBIB ------- OBIB, indented with text labels

SBIB -------- BIB, no citations
SIBIB ------- IBIB, no citations

HIT --------- Fields containing hit terms
HITIND ------ IC, ICA, ICI, NCL, CC and index field (ST and IT)
              containing hit terms
HITRN ------- HIT RN and its text modification
HITSTR ------ HIT RN, its text modification, its CA index name, and
              its structure diagram
HITSEQ ------ HIT RN, its text modification, its CA index name, its
              structure diagram, plus NTE and SEQ fields
FHITSTR ----- First HIT RN, its text modification, its CA index name, and
              its structure diagram
FHITSEQ ----- First HIT RN, its text modification, its CA index name, its
              structure diagram, plus NTE and SEQ fields
KWIC -------- Hit term plus 20 words on either side
OCC --------- Number of occurrence of hit term and field in which it occurs

To display a particular field or fields, enter the display field
codes.  For a list of the display field codes, enter HELP DFIELDS at
an arrow prompt (=>).  Examples of formats include: TI; TI,AU; BIB,ST;
TI,IND; TI,SO.  You may specify the format fields in any order and the
information will be displayed in the same order as the format
specification.

All of the formats (except for SAM, SCAN, HIT, HITIND, HITRN, HITSTR,
FHITSTR, HITSEQ, FHITSEQ, KWIC, and OCC) may be used with DISPLAY ACC
to view a specified Accession Number.
ENTER DISPLAY FORMAT (BIB):end

=> d ibib abs hitstr 19 1-66
```

L9 ANSWER 1 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 2005:298671 CAPLUS Full-text
DOCUMENT NUMBER: 144:6714
TITLE: Synthesis and reactivity of 2-polyfluoroalkylchromene-4(4H)-thiones
AUTHOR(S): Usachev, B. I.; Shafeev, M. A.; Sosnovskikh, V. Ya.
CORPORATE SOURCE: A. M. Gorky Ural State University, Yekaterinburg, 620083, Russia
SOURCE: Russian Chemical Bulletin (2004), 53(10), 2285-2292
CODEN: RCBUEY; ISSN: 1066-5285
PUBLISHER: Springer Science+Business Media, Inc.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 144:6714

AB 2-Polyfluoroalkylchromene-4(4H)-thiones, synthesized from 2-polyfluoroalkylchromones and P2S5, react with aniline, phenylhydrazines, and hydroxylamine at the C(4) atom and afford corresponding anils, phenylhydrazones, and oximes of chromones. On heating in alc. in the presence of concentrated HCl, chromone phenylhydrazones and oximes undergo ring closure to form 3-(2-hydroxyaryl)-1-phenyl-5-polyfluoroalkylpyrazoles and 5-hydroxy-3-(2-hydroxyaryl)-5-polyfluoroalkyl-Δ2-isoxazolines.

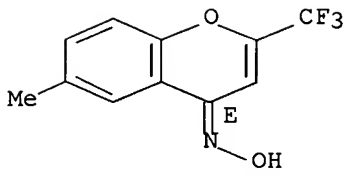
IT 869986-20-9P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of chromone anils, phenylhydrazones, and oximes via (fluoroalkyl)chromenethiones and subsequent ring closure)
RN 869986-20-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(trifluoromethyl)-, oxime, (4E)- (9CI) (CA INDEX NAME)

Double bond geometry as shown.



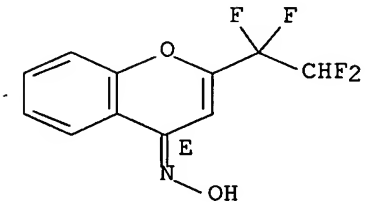
IT 869986-22-1P 869986-24-3P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of chromone anils, phenylhydrazones, and oximes via (fluoroalkyl)chromenethiones and subsequent ring closure)
RN 869986-22-1 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-methyl-2-(trifluoromethyl)-, oxime, (4E)- (9CI) (CA INDEX NAME)

Double bond geometry as shown.



RN 869986-24-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(1,1,2,2-tetrafluoroethyl)-, oxime, (4E)- (9CI) (CA INDEX NAME)

Double bond geometry as shown.



REFERENCE COUNT: 47 THERE ARE 47 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 2 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 2004:825133 CAPLUS Full-text
DOCUMENT NUMBER: 141:332051
TITLE: Preparation of substituted chromen-4-one oximes as inhibitors of protein kinases
INVENTOR(S): Green, Jeremy; Aronov, Alex; Pierce, Albert C.
PATENT ASSIGNEE(S): USA
SOURCE: U.S. Pat. Appl. Publ., 47 pp.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004198750	A1	20041007	US 2004-808678	20040325
AU 2004230841	A1	20041028	AU 2004-230841	20040325
CA 2522595	A1	20041028	CA 2004-2522595	20040325
WO 2004092154	A1	20041028	WO 2004-US9145	20040325

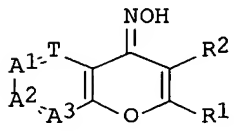
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI,

SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

EP 1615906	A1	20060118	EP 2004-758959	20040325
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, PL, SK				
JP 2006522124	T	20060928	JP 2006-509283	20040325
PRIORITY APPLN. INFO.:			US 2003-460042P	P 20030403
			WO 2004-US9145	W 20040325

OTHER SOURCE(S): MARPAT 141:332051

GI



I

AB The title compds. [I; R1 = LmR, LmAr1, LmCy1; L = S, O, NR, alkylidene wherein up to two non-adjacent methylene units of L are optionally replaced by S, O, CO, etc.; m = 0-1; Ar1 = (un)substituted 5-7 membered monocyclic or 8-10 membered bicyclic ring having 0-5 heteroatoms; Cy1 = (un)substituted 3-7 membered (un)saturated monocyclic ring having 0-3 heteroatoms or 8-10 membered (un)saturated bicyclic ring having 0-5 heteroatoms; R = H, alkyl; R2 = H, CN, SR, OR, etc.; T = N, CR3; A1-A3 = N, CR4; provided that no more than two of T, A1-A3 are N atom; R3 = H, halo, NO2, etc.; R4 = halo, NO2, CN, etc.; with provisos], useful as inhibitors of protein kinases, were prepared E.g., a 2-step synthesis of 2-(4-methoxyphenyl)-8-methylchromen-4-one oxime, starting from 8-methyl-4'-methoxyflavone, was given. The exemplified compds. I were tested and found to inhibit CDK-2, cMET, GSK-3, SYK, ZAP-70, FLT-3, JAK-3, p70S6K, TAK-1, and IRAK-4. The invention also provides pharmaceutically acceptable compns. comprising said compds. I and methods of using the compns. in the treatment of various disease, conditions, or disorders.

IT 59835-92-6P 115663-23-5P 140885-79-6P
304691-31-4P 321976-78-7P 769948-78-9P
769948-79-0P 769948-80-3P 769948-81-4P
769948-82-5P 769948-83-6P 769948-84-7P
769948-85-8P 769948-86-9P 769948-87-0P
769948-88-1P 769948-89-2P 769948-90-5P
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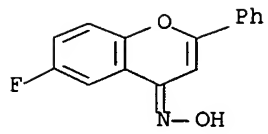
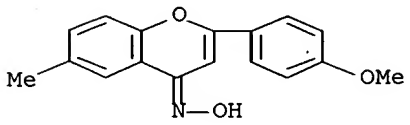
769948-97-2P 769948-98-3P 769948-99-4P
769949-00-0P 769949-01-1P 769949-02-2P
769949-03-3P 769949-04-4P 769949-06-6P
769949-07-7P 769949-08-8P 769949-09-9P
769949-10-2P 769949-11-3P 769949-12-4P
769949-13-5P 769949-14-6P 769949-15-7P
769949-16-8P 769949-17-9P
RL: PAC (Pharmacological activity); SPN (Synthetic preparation); THU (Therapeutic use); BIOL (Biological study); PREP (Preparation); USES (Uses)
(preparation of substituted chromen-4-one oximes as inhibitors of protein kinases)

RN 59835-92-6 CAPLUS

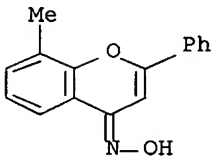
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-phenyl-, oxime (9CI) (CA INDEX NAME)



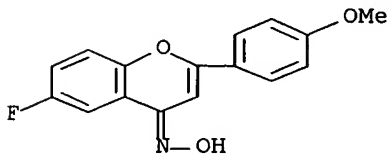
RN 115663-23-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-6-methyl-, oxime (9CI) (CA INDEX NAME)



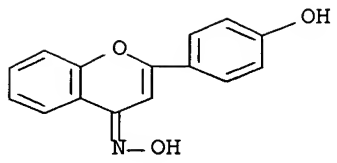
RN 140885-79-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 8-methyl-2-phenyl-, oxime (9CI) (CA INDEX NAME)



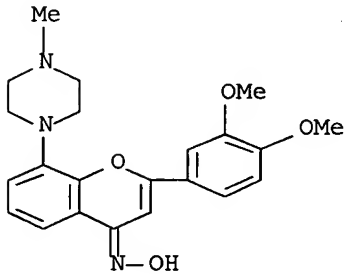
RN 304691-31-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-(4-methoxyphenyl)-, oxime (9CI) (CA INDEX NAME)



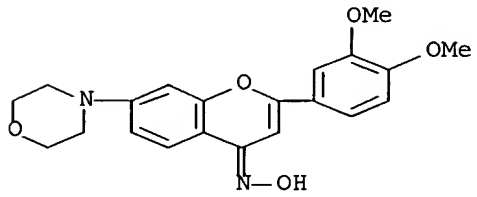
RN 321976-78-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-, oxime (9CI) (CA INDEX NAME)



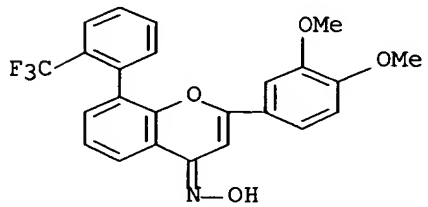
RN 769948-78-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-8-(4-methyl-1-piperazinyl)-, oxime (9CI) (CA INDEX NAME)



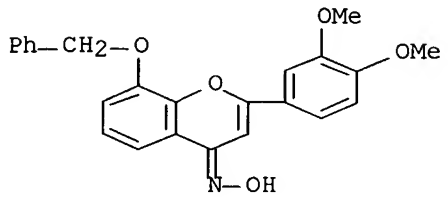
RN 769948-79-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-7-(4-morpholinyl)-, oxime (9CI) (CA INDEX NAME)



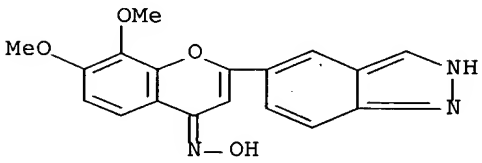
RN 769948-80-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-8-[2-(trifluoromethyl)phenyl]-, oxime (9CI) (CA INDEX NAME)



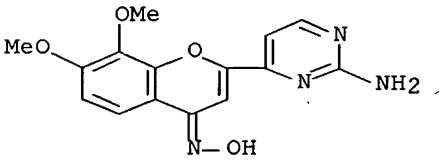
RN 769948-81-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-8-(phenylmethoxy)-, oxime (9CI) (CA INDEX NAME)



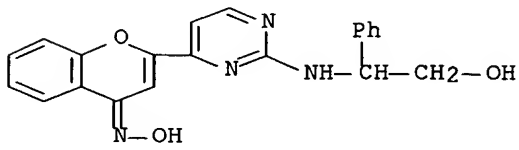
RN 769948-82-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2H-indazol-5-yl)-7,8-dimethoxy-, oxime (9CI) (CA INDEX NAME)



RN 769948-83-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2-amino-4-pyrimidinyl)-7,8-dimethoxy-, oxime (9CI) (CA INDEX NAME)

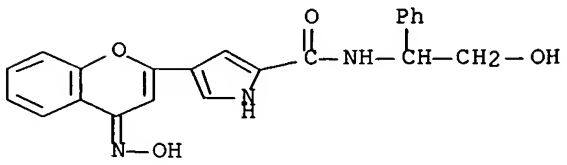


RN 769948-84-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-[2-[(2-hydroxy-1-phenylethyl)amino]-4-pyrimidinyl]-, oxime (9CI) (CA INDEX NAME)

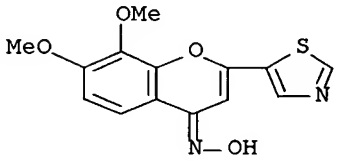


RN 769948-85-8 CAPLUS

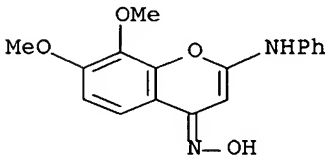
CN 1H-Pyrrole-2-carboxamide, 4-[4-(hydroxyimino)-4H-1-benzopyran-2-yl]-N-(2-hydroxy-1-phenylethyl)- (9CI) (CA INDEX NAME)



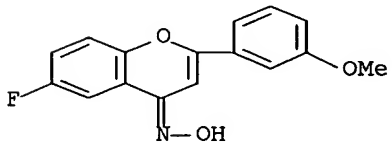
RN 769948-86-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 7,8-dimethoxy-2-(5-thiazolyl)-, oxime (9CI) (CA INDEX NAME)



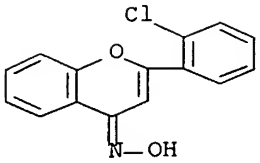
RN 769948-87-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 7,8-dimethoxy-2-(phenylamino)-, oxime (9CI) (CA INDEX NAME)



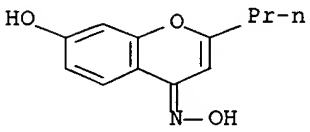
RN 769948-88-1 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-(3-methoxyphenyl)-, oxime (9CI) (CA INDEX NAME)



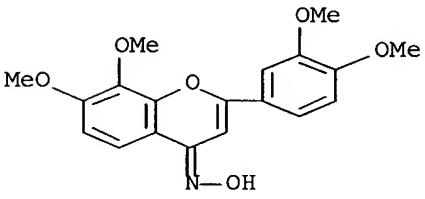
RN 769948-89-2 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2-chlorophenyl)-, oxime (9CI) (CA INDEX NAME)



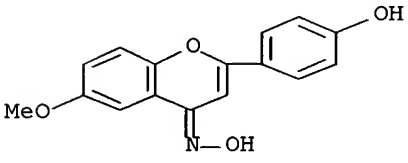
RN 769948-90-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-hydroxy-2-propyl-, oxime (9CI) (CA INDEX NAME)



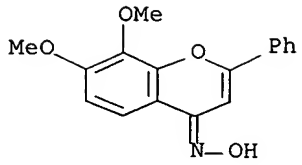
RN 769948-91-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-7,8-dimethoxy-, oxime (9CI) (CA INDEX NAME)



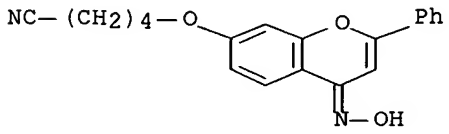
RN 769948-92-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-6-methoxy-, oxime (9CI) (CA INDEX NAME)



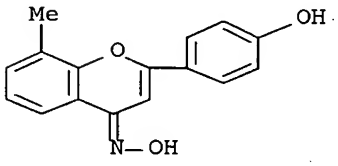
RN 769948-93-8 CAPLUS
CN 4H-1-Benzopyran-4-one, 7,8-dimethoxy-2-phenyl-, oxime (9CI) (CA INDEX NAME)



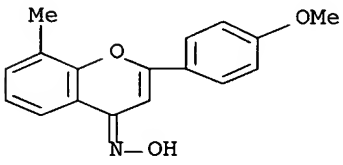
RN 769948-94-9 CAPLUS
CN Pentanenitrile, 5-[[4-(hydroxyimino)-2-phenyl-4H-1-benzopyran-7-yl]oxy]- (9CI) (CA INDEX NAME)



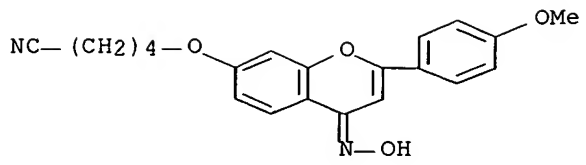
RN 769948-95-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-8-methyl-, oxime (9CI) (CA INDEX NAME)



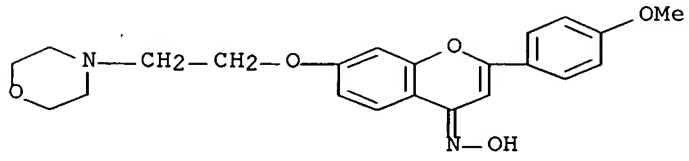
RN 769948-96-1 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-8-methyl-, oxime (9CI) (CA INDEX NAME)



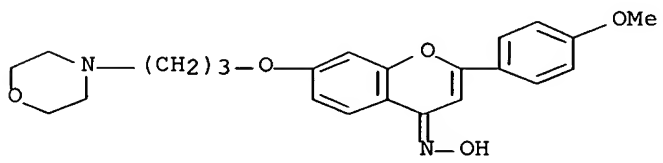
RN 769948-97-2 CAPLUS
CN Pentanenitrile, 5-[[4-(hydroxyimino)-2-(4-methoxyphenyl)-4H-1-benzopyran-7-yl]oxy]- (9CI) (CA INDEX NAME)



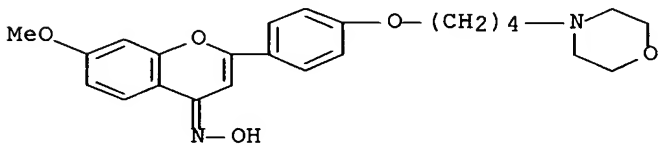
RN 769948-98-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[2-(4-morpholinyl)ethoxy]-, oxime (9CI) (CA INDEX NAME)



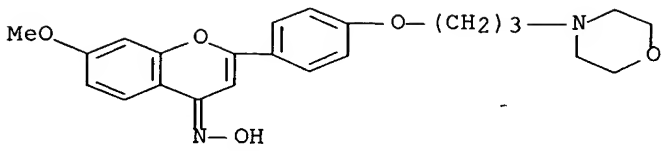
RN 769948-99-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[3-(4-morpholinyl)propoxy]-, oxime (9CI) (CA INDEX NAME)



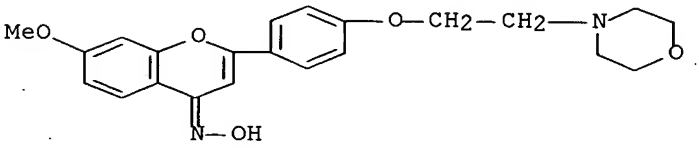
RN 769949-00-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[4-(4-morpholinyl)butoxy]phenyl]-, oxime (9CI) (CA INDEX NAME)



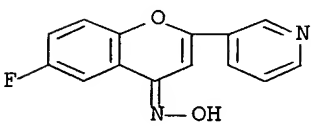
RN 769949-01-1 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[3-(4-morpholinyl)propoxy]phenyl]-, oxime (9CI) (CA INDEX NAME)



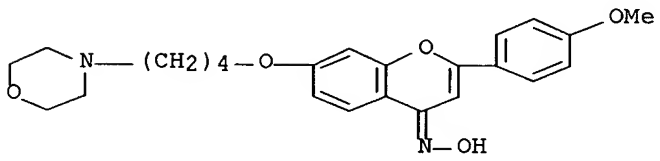
RN 769949-02-2 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[2-(4-morpholinyl)ethoxy]phenyl]-, oxime (9CI) (CA INDEX NAME)



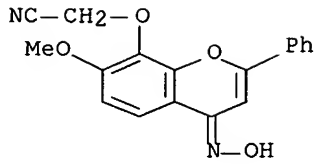
RN 769949-03-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-(3-pyridinyl)-, oxime (9CI) (CA INDEX NAME)



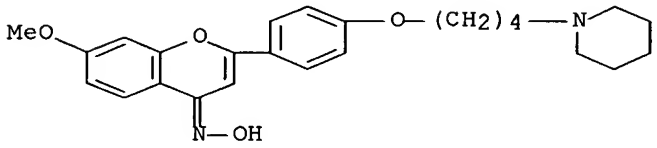
RN 769949-04-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[4-(4-morpholinyl)butoxy]-, oxime (9CI) (CA INDEX NAME)



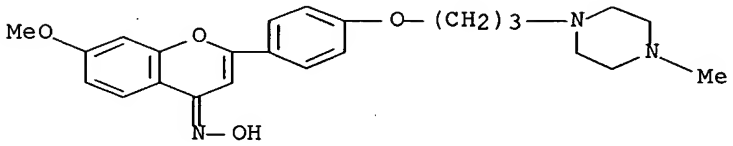
RN 769949-06-6 CAPLUS
CN Acetonitrile, [[4-(hydroxyimino)-7-methoxy-2-phenyl-4H-1-benzopyran-8-yl]oxy]- (9CI) (CA INDEX NAME)



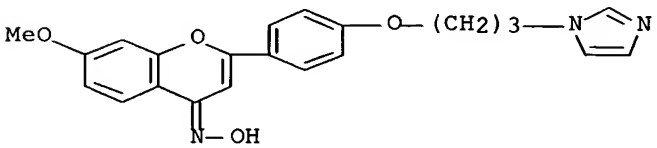
RN 769949-07-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[4-(1-piperidinyl)butoxy]phenyl]-, oxime (9CI) (CA INDEX NAME)



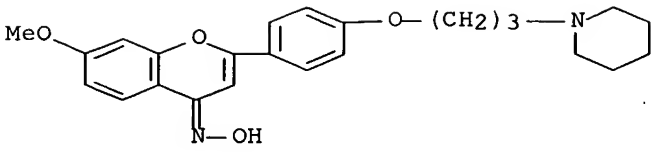
RN 769949-08-8 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[3-(4-methyl-1-piperazinyl)propoxy]phenyl]-, oxime (9CI) (CA INDEX NAME)



RN 769949-09-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-[4-[3-(1H-imidazol-1-yl)propoxy]phenyl]-7-methoxy-, oxime (9CI) (CA INDEX NAME)



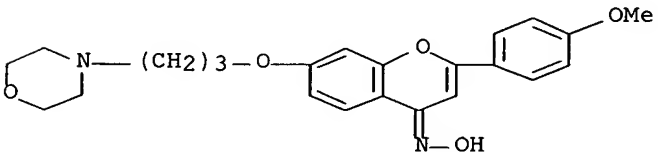
RN 769949-10-2 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[3-(1-piperidinyl)propoxy]phenyl]-, oxime (9CI) (CA INDEX NAME)



RN 769949-11-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[3-(4-morpholinyl)propoxy]-, oxime, mono(trifluoroacetate) (9CI) (CA INDEX NAME)

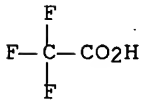
CM 1

CRN 769948-99-4
CMF C23 H26 N2 O5



CM 2

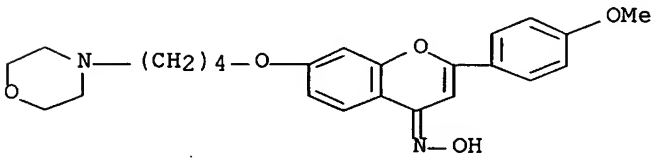
CRN 76-05-1
CMF C2 H F3 O2



RN 769949-12-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[4-(4-morpholinyl)butoxy]-, oxime, mono(trifluoroacetate) (9CI) (CA INDEX NAME)

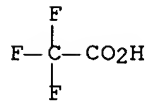
CM 1

CRN 769949-04-4
CMF C24 H28 N2 O5



CM 2

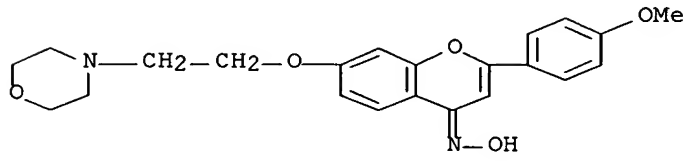
CRN 76-05-1
CMF C2 H F3 O2



RN 769949-13-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[2-(4-morpholinyl)ethoxy]-, oxime, mono(trifluoroacetate) (9CI) (CA INDEX NAME)

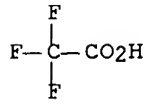
CM 1

CRN 769948-98-3
CMF C22 H24 N2 O5



CM 2

CRN 76-05-1
CMF C2 H F3 O2

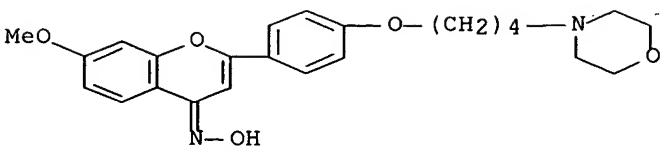


RN 769949-14-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[4-(4-morpholinyl)butoxy]phenyl]-, oxime, mono(trifluoroacetate) (9CI) (CA INDEX NAME)

CM 1

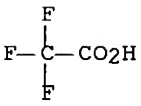
CRN 769949-00-0

CMF C24 H28 N2 O5



CM 2

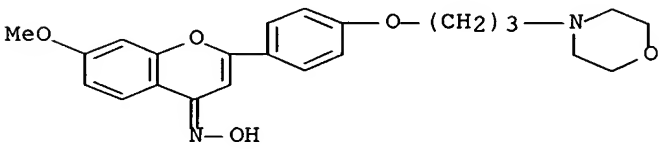
CRN 76-05-1
CMF C2 H F3 O2



RN 769949-15-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[3-(4-morpholinyl)propoxy]phenyl]-, oxime, mono(trifluoroacetate) (9CI) (CA INDEX NAME)

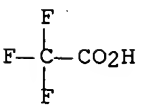
CM 1

CRN 769949-01-1
CMF C23 H26 N2 O5



CM 2

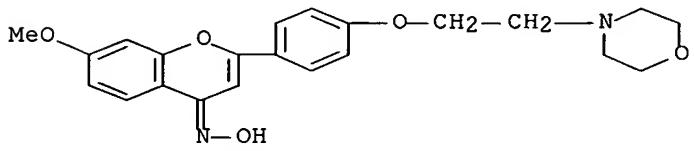
CRN 76-05-1
CMF C2 H F3 O2



RN 769949-16-8 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[2-(4-morpholinyl)ethoxy]phenyl]-, oxime, mono(trifluoroacetate) (9CI) (CA INDEX NAME)

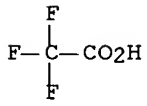
CM 1

CRN 769949-02-2
CMF C22 H24 N2 O5



CM 2

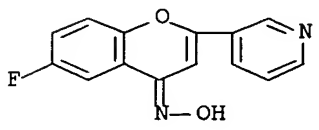
CRN 76-05-1
CMF C2 H F3 O2



RN 769949-17-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-(3-pyridinyl)-, oxime, mono(trifluoroacetate) (9CI) (CA INDEX NAME)

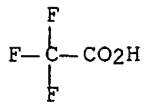
CM 1

CRN 769949-03-3
CMF C14 H9 F N2 O2



CM 2

CRN 76-05-1
CMF C2 H F3 O2

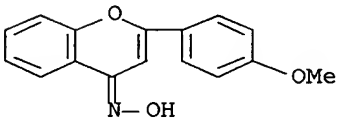


L9 ANSWER 3 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 2002:785496 CAPLUS Full-text
DOCUMENT NUMBER: 138:170044
TITLE: Amino acid derivatives and oximes of flavones
AUTHOR(S): Ishchenko, V. V.; Shulyak, T. S.; Khilya, V. P.
CORPORATE SOURCE: Taras Shevchenko Kiev National University, Kiev, Ukraine
SOURCE: Chemistry of Heterocyclic Compounds (New York, NY, United States)(Translation of Khimiya Geterotsiklicheskikh Soedinenii) (2002), 38(3), 274-280
CODEN: CHCCAL; ISSN: 0009-3122
PUBLISHER: Kluwer Academic/Consultants Bureau
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:170044

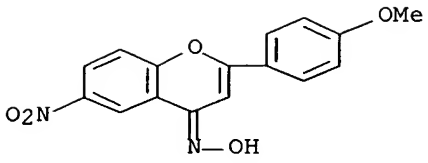
AB 4-Ethoxyflavylium tetrafluoroborates with substituents in rings A and B were synthesized. Their reaction with nitrogen-containing nucleophiles was investigated. It was shown that derivs. of flavones at the carbonyl group are formed as a result of these reactions. The major distinctive physicochem. characteristics of the oximes of flavones and isoxazoles were determined For example, the reaction of flavylium perchlorate with hydroxylamine hydrochloride gave 2-(3-phenyl-5-isoxazolyl)phenol.

IT 82340-45-2P, 2-(4-Methoxyphenyl)-4H-1-Benzopyran-4-one oxime
300835-66-9P, 2-(4-Methoxyphenyl)-6-nitro-4H-1-Benzopyran-4-one oxime 463358-39-6P, 6-Bromo-2-(4-methoxyphenyl)-4H-1-Benzopyran-4-one oxime
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of 2-phenyl-4H-1-benzopyran-4-one oximes and N-(2-phenyl-4H-1-benzopyran-4-ylidene) amino acid derivs. from 4-ethoxy-2-phenylbenzopyrylium tetrafluoroborates)

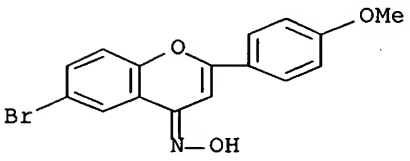
RN 82340-45-2 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-, oxime (9CI) (CA INDEX NAME)



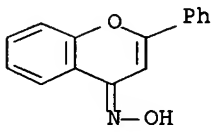
RN 300835-66-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-6-nitro-, oxime (9CI) (CA INDEX NAME)



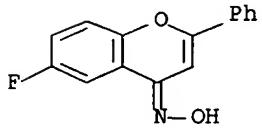
RN 463358-39-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-bromo-2-(4-methoxyphenyl)-, oxime (9CI) (CA INDEX NAME)



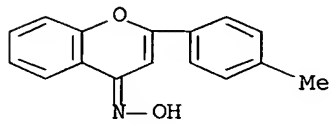
IT 22115-89-5P, 2-Phenyl-4H-1-Benzopyran-4-one oxime
59835-92-6P, 6-Fluoro-2-(phenyl)-4H-1-Benzopyran-4-one oxime
63645-49-8P, 2-(4-Methylphenyl)-4H-1-Benzopyran-4-one oxime
135085-53-9P, 6-Chloro-2-(4-methoxyphenyl)-4H-1-Benzopyran-4-one oxime 304691-31-4P, 6-Fluoro-2-(4-methoxyphenyl)-4H-1-Benzopyran-4-one oxime 321976-78-7P, 2-(4-Hydroxyphenyl)-4H-1-Benzopyran-4-one oxime 321976-79-8P, 6-Fluoro-2-(4-hydroxyphenyl)-4H-1-Benzopyran-4-one oxime 321976-80-1P, 2-(4-Hydroxyphenyl)-6-nitro-4H-1-Benzopyran-4-one oxime 497869-41-7P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of 2-phenyl-4H-1-benzopyran-4-one oximes and N-(2-phenyl-4H-1-benzopyran-4-ylidene) amino acid derivs. from 4-ethoxy-2-phenylbenzopyrylium tetrafluoroborates)
RN 22115-89-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)



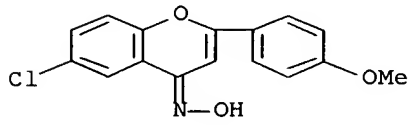
RN 59835-92-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-phenyl-, oxime (9CI) (CA INDEX NAME)



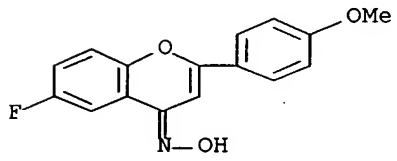
RN 63645-49-8 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methylphenyl)-, oxime (9CI) (CA INDEX NAME)



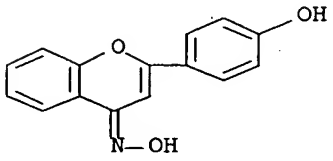
RN 135085-53-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-chloro-2-(4-methoxyphenyl)-, oxime (9CI) (CA INDEX NAME)



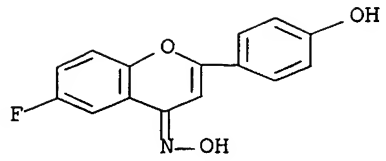
RN 304691-31-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-(4-methoxyphenyl)-, oxime (9CI) (CA INDEX NAME)



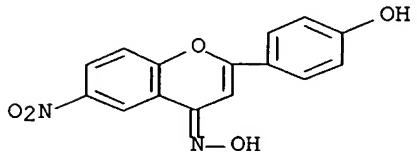
RN 321976-78-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-, oxime (9CI) (CA INDEX NAME)



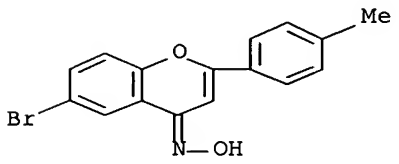
RN 321976-79-8 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-(4-hydroxyphenyl)-, oxime (9CI) (CA INDEX NAME)



RN 321976-80-1 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-6-nitro-, oxime (9CI) (CA INDEX NAME)



RN 497869-41-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-bromo-2-(4-methylphenyl)-, oxime (9CI) (CA INDEX NAME)



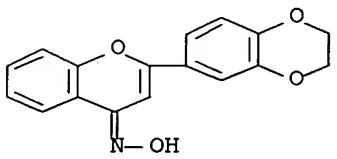
REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 4 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 2001:175148 CAPLUS Full-text
DOCUMENT NUMBER: 134:326293
TITLE: Synthetic analogs of naturally occurring flavolignans. X. Reaction of flavones and their thioderivatives with hydroxylamine
AUTHOR(S): Aitmambetov, A.; Khilya, V. P.; Kubzheterova, A.
CORPORATE SOURCE: Complex Institute of Natural Sciences, Karakalpak Division, Academy of Sciences of the Republic of Uzbekistan, Nukus, 742000, Uzbekistan
SOURCE: Chemistry of Natural Compounds (Translation of Khimiya Prirodnykh Soedinenii) (2000), 36(1), 47-50
CODEN: CHNCA8; ISSN: 0009-3130
PUBLISHER: Consultants Bureau
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:326293
AB 1,3-Benzodioxoles, 1,4-benzodioxanes, and 1,5-benzodioxepanes are flavone analogs that hydroxylamine recyclizes into derivs. of 5-(2-

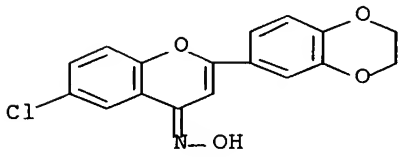
hydroxyphenyl)isoxazoles. They react with thio derivs. with retention of the pyrone ring and formation of oximes. Their structures are proven using PMR spectra.

IT 168788-23-6P 302935-66-6P
RL: SPN (Synthetic preparation); PREP (Preparation)
(reaction of hydroxylamine with flavones and their thio derivs.)

RN 168788-23-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2,3-dihydro-1,4-benzodioxin-6-yl)-, oxime (9CI) (CA INDEX NAME)



RN 302935-66-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-chloro-2-(2,3-dihydro-1,4-benzodioxin-6-yl)-, oxime (9CI) (CA INDEX NAME)



REFERENCE COUNT: 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 5 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2000:378390 CAPLUS Full-text
DOCUMENT NUMBER: 133:129525
TITLE: Properties and activity screening of chromone derivatives
AUTHOR(S): Kim, Young Ro; Lee, Sang Heyn; Kim, Kyoung Soon; Cheong, Chun Sik; Cheong, Jae Hoon; Kim, Bak Kwang
CORPORATE SOURCE: College of Pharmacy, Seoul National University, Seoul, 152-742, S. Korea
SOURCE: Yakhak Hoechi (2000), 44(2), 107-114
CODEN: YAHOA3; ISSN: 0513-4234
PUBLISHER: Pharmaceutical Society of Korea
DOCUMENT TYPE: Journal
LANGUAGE: Korean

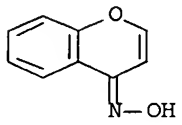
AB We have synthesized the chromone derivs. 4-isonitroso-4H-1-benzopyran and 4-amino-2,3-dihydro-4H-1-benzopyran by using a condensation method. Physicochem. properties of these compds. were measured and analyzed by UV and HPLC method. The correlation coefficient of their methanol solns. by UV were 0.9992 and 0.9994, resp. An oxime compound was resolved within 4 min and had a detection limit of 3 ng at S/N=3 by HPLC using a reversed phase column with three solvents (MeOH, H2O, HAc). The amino compound was resolved within 4.5 min and had a detection limit of 10 ng at S/N=3 by HPLC under the same conditions.

Anti-diabetic effect of chromone derivs. were investigated in the streptozotocin (STZ)-induced diabetic rats. Diabetes was induced in male Sprague-Dawley rats by injections of STZ (45 mg/kg, i.v.). The investigation of the hair growth effect of isonitrosobenzopyran and 4-aminobenzopyran on the hair of black mice (C57BL/6) was also carried out. The administration of their ethanol solution to the black mouse (C57BV/6) through skin promoted the growth of hair.

IT 61348-46-7P
RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); THU (Therapeutic use); BIOL (Biological study); PREP (Preparation); USES (Uses)
(properties and activity screening of chromone derivs.)

RN 61348-46-7 CAPLUS

CN 4H-1-Benzopyran-4-one, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 6 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1998:290410 CAPLUS Full-text

DOCUMENT NUMBER: 129:4344

TITLE: Evaluation of effect of microwave irradiation on syntheses and reactions of some new 3-acyl-methylchromones

AUTHOR(S): Lacova, Margita; El-Shaaer, Hafez M.; Loos, Dusan; Matulova, Maria; Chovancova, Jarmila; Furdik, Mikulas

CORPORATE SOURCE: Department Organic Chemistry, Faculty Natural Sciences, Comenius University, Bratislava, SK-842 15, Slovakia

SOURCE: Molecules [Electronic Publication] (1998), 3(3), 120-131
CODEN: MOLEFW; ISSN: 1420-3049
URL: http://mdpi.org./molecules/papers/30300120.pdf

PUBLISHER: Molecular Diversity Preservation International

DOCUMENT TYPE: Journal; (online computer file)

LANGUAGE: English

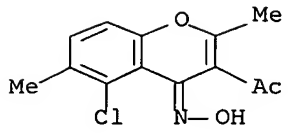
OTHER SOURCE(S): CASREACT 129:4344

AB 3-Acyl-2-R-methylchromones [R = H, ArO, C6H4(CO)2N] were prepared in good yields by different methods from (2-hydroxyaroyl)acetone derivs. Some subsequent reactions of these compds. with HONH2 and 3-formylchromones are described. The effect of microwave irradiation on some condensation reactions was studied.

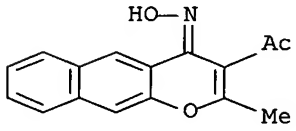
IT 207387-87-9P 207387-88-0P 207387-89-1P
207387-90-4P 207387-91-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(evaluation of effect of microwave irradiation on syntheses and reactions of some new acylmethylchromones)

RN 207387-87-9 CAPLUS

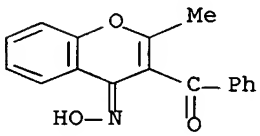
CN 4H-1-Benzopyran-4-one, 3-acetyl-5-chloro-2,6-dimethyl-, 4-oxime (9CI) (CA INDEX NAME)



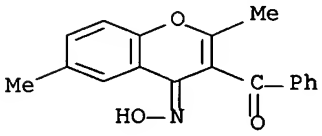
RN 207387-88-0 CAPLUS
CN 4H-Naphtho[2,3-b]pyran-4-one, 3-acetyl-2-methyl-, 4-oxime (9CI) (CA INDEX NAME)



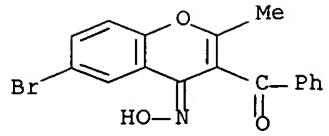
RN 207387-89-1 CAPLUS
CN 4H-1-Benzopyran-4-one, 3-benzoyl-2-methyl-, 4-oxime (9CI) (CA INDEX NAME)



RN 207387-90-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 3-benzoyl-2,6-dimethyl-, 4-oxime (9CI) (CA INDEX NAME)



RN 207387-91-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 3-benzoyl-6-bromo-2-methyl-, 4-oxime (9CI) (CA INDEX NAME)

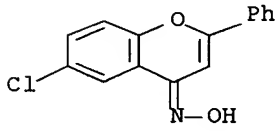


L9 ANSWER 7 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1996:512488 CAPLUS Full-text
DOCUMENT NUMBER: 125:221509
TITLE: A comparative study-reaction of chalcones with hydroxylamine hydrochloride in different solvents
AUTHOR(S): Rahatgaonkar, A.M.; Ghiya, B.J.
CORPORATE SOURCE: Chemistry Department, Institute of Science, Nagpur, 440 001, India
SOURCE: Indian Journal of Heterocyclic Chemistry (1996), 5(4), 323-324
CODEN: IJCHEI; ISSN: 0971-1627
PUBLISHER: Lucknow University, Dep. of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English

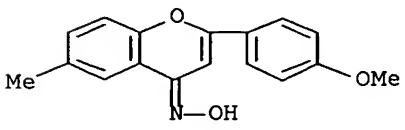
AB A comparative study of the reaction between chalcones and hydroxylamine hydrochloride was found to be interesting as time of reaction, yield and the nature of products are dependent not only on the substituents in chalcones but the type of the solvent used for reaction medium. The following solvents/solvent reagent mixture were considered as reaction medium, S1 (Ethanol & sodium acetate) S2 (DMF), S3 (DMSO), S4 (DMSO-I2), S5 (Acetic Acid) S6 (Ethanol & KOH). The products are flavanone oximes, flavone oximes or isoxazolines. Time varied from 10 min to 1 h for the maximum possible yield of a single particular compound out of three mentioned.

IT 59835-93-7P 115663-23-5P 115663-26-8P
135085-53-9P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

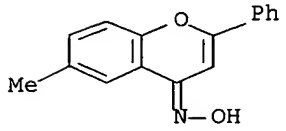
RN 59835-93-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-chloro-2-phenyl-, oxime (9CI) (CA INDEX NAME)



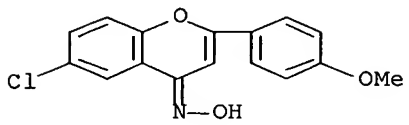
RN 115663-23-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-6-methyl-, oxime (9CI) (CA INDEX NAME)



RN 115663-26-8 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-methyl-2-phenyl-, oxime (9CI) (CA INDEX NAME)



RN 135085-53-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-chloro-2-(4-methoxyphenyl)-, oxime (9CI) (CA INDEX NAME)

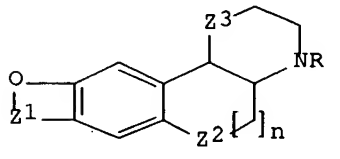


L9 ANSWER 8 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1996:177849 CAPLUS Full-text
DOCUMENT NUMBER: 124:232473
TITLE: Preparation of tetracyclic 1,4-oxazine derivatives and analogs as dopaminergic D3 agonists
INVENTOR(S): Peglion, Jean-Louis; Vian, Joel; Goument, Bertrand; Millan, Mark; Audinot, Valerie; Schwartz, Jean-Charles; Sokoloff, Pierre
PATENT ASSIGNEE(S): Adir et Compagnie, Fr.; Institute National de la Sante et de la Recherche Medicale (INSERM)
SOURCE: Eur. Pat. Appl., 21 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

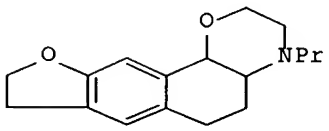
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 686637	A1	19951213	EP 1995-401311	19950607
EP 686637	B1	20000426		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, MC, NL, PT, SE				
FR 2721027	A1	19951215	FR 1994-6985	19940608
FR 2721027	B1	19960719		
US 5593989	A	19970114	US 1995-456504	19950601
CA 2151096	A1	19951209	CA 1995-2151096	19950606
CA 2151096	C	20030708		
AU 9520523	A	19951214	AU 1995-20523	19950606
AU 681780	B2	19970904		
FI 9502802	A	19951209	FI 1995-2802	19950607

FI 111948	B1	20031015			
NO 9502249	A	19951211	NO 1995-2249		19950607
CN 1120541	A	19960417	CN 1995-107344		19950607
CN 1050358	B	20000315			
AT 192155	T	20000515	AT 1995-401311		19950607
PT 686637	T	20000831	PT 1995-401311		19950607
ES 2147825	T3	20001001	ES 1995-401311		19950607
JP 07330778	A	19951219	JP 1995-141772		19950608
JP 3157418	B2	20010416			
ZA 9504738	A	19960126	ZA 1995-4738		19950608
US 5668142	A	19970916	US 1996-659267		19960606
GR 3033762	T3	20001031	GR 2000-401458		20000623
PRIORITY APPLN. INFO.:			FR 1994-6985	A	19940608
			US 1995-456504	A3	19950601

OTHER SOURCE(S): MARPAT 124:232473
GI



I



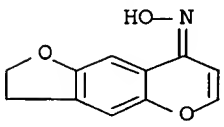
II

AB Title compds. [I; R = H, (ar)alk(en)yl, (ar)alkynyl, etc.; Z1 = (CH2)2-3, CH:CH, CH2CO, CH2CH(OH); Z2,Z3 = O or CH2; n = 0 or 1] were prepared Thus, 7-amino-2,3,6,7-tetrahydro-5H-naphtho[2,3-b]furan-8-one was converted in 6 steps to title compound trans-II which reduced immobility from 188.0s (control) to 63.1s at 2.5mg/kg s.c. in the forced swimming test (test animal not given).

IT 174637-13-9P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of tetracyclic 1,4-oxazine derivs. and analogs as dopaminergic D3 agonists)

RN 174637-13-9 CAPLUS

CN 8H-Furo[2,3-g][1]benzopyran-8-one, 2,3-dihydro-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 9 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

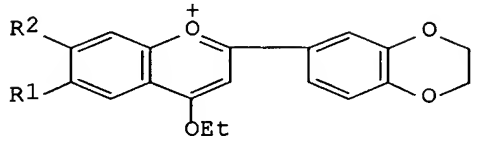
ACCESSION NUMBER: 1995:603160 CAPLUS Full-text

DOCUMENT NUMBER: 123:256619

TITLE: Dioxane analogs of flavylium salts

AUTHOR(S): Ishchenko, V. V.; Nosichenko, E. I.; Falkovskaya, O.

T.; Khilya, V. P.
CORPORATE SOURCE: Kiev. Univ., Ukraine
SOURCE: Khimiya Geterotsiklicheskikh Soedinenii (1995), (3), 322-4
CODEN: KGSSAQ; ISSN: 0132-6244
PUBLISHER: Latviiskii Institut Organicheskogo Sinteza
DOCUMENT TYPE: Journal
LANGUAGE: Russian
GI

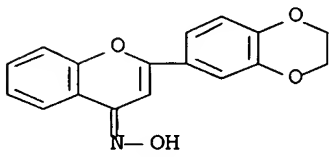


I

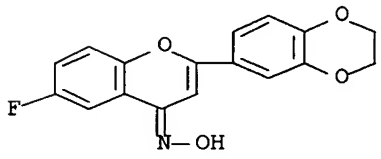
AB The title compds. (I; R1 = H, F, Me; R2 = H, Me, MeO) were prepared, and their reactions with hydrazine, hydroxylamine, aniline, and phenylhydrazine.

IT 168788-23-6P 168788-24-7P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

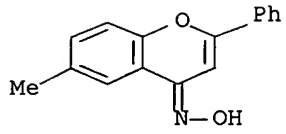
RN 168788-23-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2,3-dihydro-1,4-benzodioxin-6-yl)-, oxime (9CI) (CA INDEX NAME)



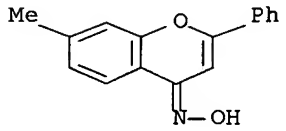
RN 168788-24-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2,3-dihydro-1,4-benzodioxin-6-yl)-6-fluoro-, oxime (9CI) (CA INDEX NAME)



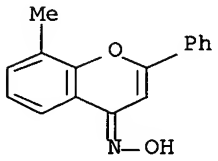
L9 ANSWER 10 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1992:214399 CAPLUS Full-text



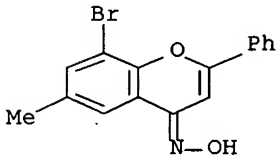
RN 140885-78-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methyl-2-phenyl-, oxime (9CI) (CA INDEX NAME)



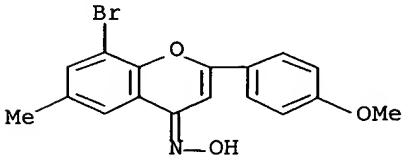
RN 140885-79-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 8-methyl-2-phenyl-, oxime (9CI) (CA INDEX NAME)



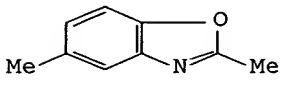
RN 140885-80-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 8-bromo-6-methyl-2-phenyl-, oxime (9CI) (CA INDEX NAME)



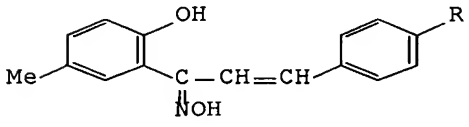
RN 140885-81-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 8-bromo-2-(4-methoxyphenyl)-6-methyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 11 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1991:471443 CAPLUS Full-text
DOCUMENT NUMBER: 115:71443
TITLE: Reaction of oximes of 2-hydroxyacetophenone, chalcone, flavanone, and flavone
AUTHOR(S): Bagade, M. B.; Ghiya, B. J.
CORPORATE SOURCE: Dep. Org. Chem., Inst. Sci., Nagpur, 440 001, India
SOURCE: Asian Journal of Chemistry (1991), 3(2), 158-63
CODEN: AJCHEW; ISSN: 0970-7077
DOCUMENT TYPE: Journal
LANGUAGE: English
GI



II

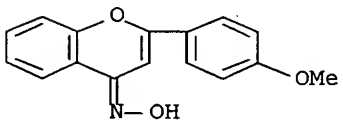


III

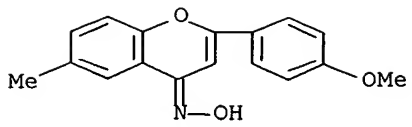
AB Oximes of 2-hydroxyacetophenone, chalcone, flavanone, and flavone were prepared by the action of hydroxylamine hydrochloride on the resp. compds. The oximes gave back the starting material by the action of HCl, nitrous acid or CrO3 in AcOH. 2-Hydroxy-5-methylacetophenone oxime (I), with POCl3, cyclized to give benzoxazole II. I also condensed with RCHO (R = Ph, 4-MeOC6H4) to give chalcone oxime III.

IT 82340-45-2P 115663-23-5P 135085-53-9P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and intramol. cyclocondensation of)

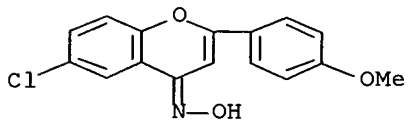
RN 82340-45-2 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-, oxime (9CI) (CA INDEX NAME)



RN 115663-23-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-6-methyl-, oxime (9CI) (CA INDEX NAME)



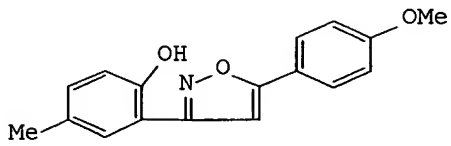
RN 135085-53-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-chloro-2-(4-methoxyphenyl)-, oxime (9CI) (CA INDEX NAME)



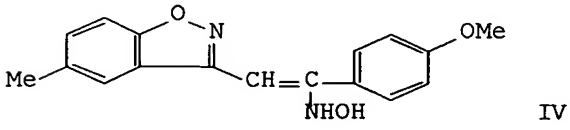
L9 ANSWER 12 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:	1988:473368 CAPLUS Full-text
DOCUMENT NUMBER:	109:73368
TITLE:	Reaction of hydroxylamine hydrochloride with 2-hydroxy-4'-methoxy-5-methyldibenzoylmethane and 4'-methoxy-6-methylflavone
AUTHOR(S):	Lohiya, S. B.; Ghiya, B. J.
CORPORATE SOURCE:	Dep. Chem., Vidarbha Mahadivyalaya, Amravati, 444 604, India
SOURCE:	Indian Journal of Chemistry, Section B: Organic Chemistry Including Medicinal Chemistry (1987), 26B(9), 873-6 CODEN: IJSBDB; ISSN: 0376-4699
DOCUMENT TYPE:	Journal
LANGUAGE:	English
OTHER SOURCE(S):	CASREACT 109:73368

GI



III



IV

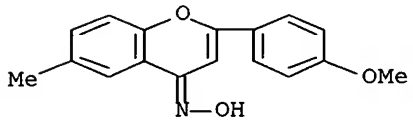
AB 2-Hydroxy-4'-methoxy-5-methyldibenzoylmethane (I) reacts with NH2OH·HCl (II) in pyridine to give a mixture of isoxazole III and benzisoxazole IV. Similar results are obtained from the reaction of I with II in ethylenediamine, aqueous DMF, pyridine in ethanol or in the presence of KOH in MeOH. However,

in DMF or in the presence of NaHCO3 in EtOH only III is formed. I remains unchanged in benzene/sodium bicarbonate and affords 4'-methoxy-6-methylflavone (V) in dilute AcOH or MeOH. V reacts with II in pyridine or ethylenediamine to give 4'-methoxy-6-methylflavone oxime instead of III as suggested by previous workers. 2-Hydroxy-5-methyldibenzoylmethane and 6-methylflavone also give similar results.

IT 115663-23-5P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and hydrolysis of)

RN 115663-23-5 CAPLUS

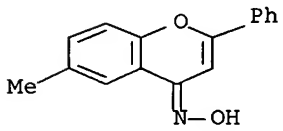
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-6-methyl-, oxime (9CI) (CA INDEX NAME)



IT 115663-26-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

RN 115663-26-8 CAPLUS

CN 4H-1-Benzopyran-4-one, 6-methyl-2-phenyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 13 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1987:176226 CAPLUS Full-text

DOCUMENT NUMBER: 106:176226

TITLE: Reaction of chromones with hydroxylamine in anhydrous methanol. A novel route for the preparation of chromone oximes

AUTHOR(S): Szabo, Vince; Borbely, Janos; Theisz, Edit; Nagy, Sandor

CORPORATE SOURCE: Dep. Appl. Chem., Kossuth Lajos Univ., Debrecen, H-4010, Hung.

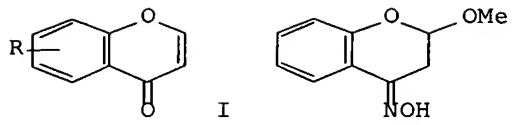
SOURCE: Tetrahedron (1986), 42(15), 4215-22
CODEN: TETRAB; ISSN: 0040-4020

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 106:176226

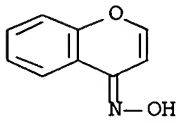
GI



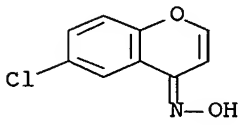
AB Chromone (I, R = H) is transformed mainly into its oxime with NH2OH.HCl in anhydrous MeOH. Isoxazole derivs. were also isolated, and the formation of 2-methoxychromanone has been detected, as well. Depending on the character of the substituent, chromones I (R = 7-OMe, 7-Cl, 7-NO2, 6-Cl, 6-NO2, 6-Me) afforded 2-methoxychromanone oximes II, chromone oximes, or isoxazolines III as the isolable major product. II, which were also prepared by an acid-catalyzed MeOH addition on I are regarded the key intermediates of the formation of chromone oximes.

IT 61348-46-7P, Chromone oxime 86176-49-0P
99184-95-9P 107826-00-6P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

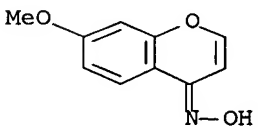
RN 61348-46-7 CAPLUS
CN 4H-1-Benzopyran-4-one, oxime (9CI) (CA INDEX NAME)



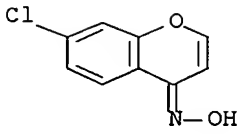
RN 86176-49-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-chloro-, oxime (9CI) (CA INDEX NAME)



RN 99184-95-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-, oxime (9CI) (CA INDEX NAME)



RN 107826-00-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-chloro-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 14 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1986:109420 CAPLUS Full-text
DOCUMENT NUMBER: 104:109420
TITLE: Reaction of benzofuranic analogs of flavone and isoflavone with nucleophilic reagents
AUTHOR(S): Grishko, L. G.; Khilya, V. P.; Sedyuko, M. F.; Litkei, D.
CORPORATE SOURCE: Kiev. Gos. Univ., Kiev, USSR
SOURCE: Ukrainskii Khimicheskii Zhurnal (Russian Edition) (1985), 51(2), 211-17
CODEN: UKZHAU; ISSN: 0041-6045
DOCUMENT TYPE: Journal
LANGUAGE: Russian
OTHER SOURCE(S): CASREACT 104:109420
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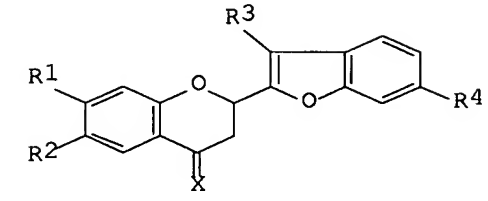

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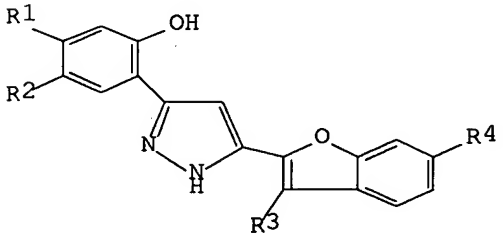
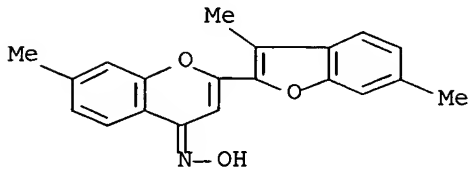


II

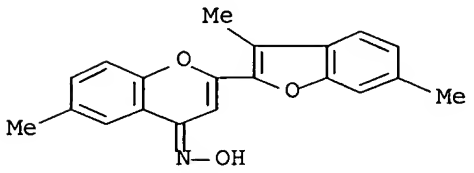
AB Treating chromone I (R1 = H, Me, R2-R4 = H; R1 = H, R2-R4 = Me; R1 = R3 = R4 = Me, R2 = H; X = O) with P2S5 in pyridine 0.5-1 h at 105-110° gave 55-90% I (X = S). Cyclization of the chromones by N2H4.H2O in refluxing EtOH 1-15 h gave 70-91% pyrazoles II. Chromone oximes I (X = NOH) were obtained in 32-96% yields by treating I (X = O) with NH2OH.HCl in pyridine 8 h at 110-115°.

IT 99819-00-8P 99819-01-9P 99819-02-0P
99819-03-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

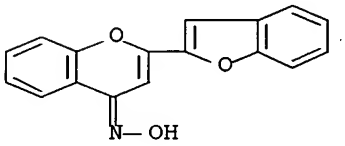
RN 99819-00-8 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,6-dimethyl-2-benzofuranyl)-7-methyl-, oxime (9CI) (CA INDEX NAME)



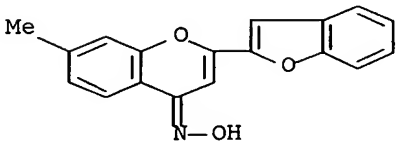
RN 99819-01-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,6-dimethyl-2-benzofuranyl)-6-methyl-, oxime (9CI) (CA INDEX NAME)



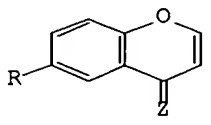
RN 99819-02-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2-benzofuranyl)-, oxime (9CI) (CA INDEX NAME)



RN 99819-03-1 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2-benzofuranyl)-7-methyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 15 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1983:421652 CAPLUS Full-text
DOCUMENT NUMBER: 99:21652
TITLE: A new pathway in the reactions of chromones and hydroxylamine in anhydrous solutions
AUTHOR(S): Szabo, Vince; Borbely, Janos; Theisz, Edit; Janzso, Geza
CORPORATE SOURCE: Inst. Appl. Chem., Kossuth Lajos Univ., Debrecen, H-4010, Hung.
SOURCE: Tetrahedron Letters (1982), 23(50), 5347-50
CODEN: TELEAY; ISSN: 0040-4039
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 99:21652
GI



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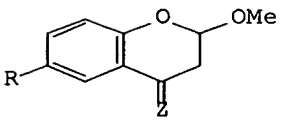
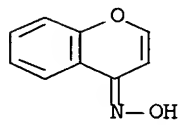


II

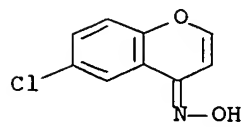
AB Reaction of chromones I (Z = O, R = H, Cl, NO2) with NH2OH.HCl in anhydrous MeOH to give oximes I (Z = NOH, R as before) and methoxychromanone oximes II (Z = NHOH, R as before) proceeds via the methoxychromanone intermediates II (Z = O, R as before).

IT 61348-46-7P 86176-49-0P 86176-50-3P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of, by reaction of chromone and hydroxylamine hydrochloride, mechanism of)

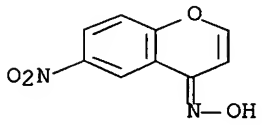
RN 61348-46-7 CAPLUS
CN 4H-1-Benzopyran-4-one, oxime (9CI) (CA INDEX NAME)



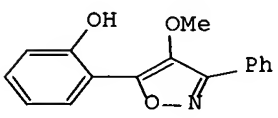
RN 86176-49-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-chloro-, oxime (9CI) (CA INDEX NAME)



RN 86176-50-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-nitro-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 16 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1983:197947 CAPLUS Full-text
DOCUMENT NUMBER: 98:197947
TITLE: Benzo-γ-pyrones. Part IX. Reaction of 3-methoxyflavone with hydroxylamine hydrochloride
AUTHOR(S): Maib, Piotr; Basinski, Wlodzimierz
CORPORATE SOURCE: Fac. Pharm., Sch. Med., Lodz, 90145, Pol.
SOURCE: Polish Journal of Chemistry (1981), 55(7-8), 1527-33
CODEN: PJCHDQ; ISSN: 0137-5083
DOCUMENT TYPE: Journal
LANGUAGE: English
GI



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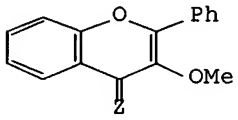
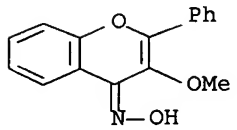


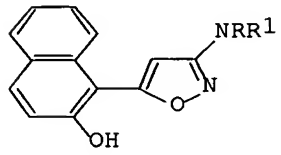
II

AB Isoxazole derivative I was obtained when 3-methoxyflavone was heated with HONH2.HCl and NaOH in EtOH. The reaction of thioflavone II (Z = S) with HONH2.HCl in pyridine gave II (Z = NOH).
IT 85726-16-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 85726-16-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 3-methoxy-2-phenyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 17 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1982:492178 CAPLUS Full-text
DOCUMENT NUMBER: 97:92178
TITLE: Reaction of 3-(dialkylamino)-1H-naphtho[2,1-b]pyran-1-one with hydroxylamine
AUTHOR(S): Balbi, A.; Ermili, A.; Roma, G.; Mazzei, M.
CORPORATE SOURCE: Ist. Sci. Farm., Univ. Genova, Genoa, Italy
SOURCE: Farmaco, Edizione Scientifica (1982), 37(6), 387-97
CODEN: FRPSAX; ISSN: 0430-0920
DOCUMENT TYPE: Journal
LANGUAGE: Italian
OTHER SOURCE(S): CASREACT 97:92178
GI



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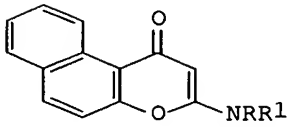
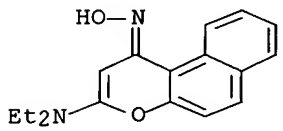


II

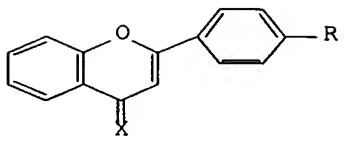
AB 5-(2-Hydroxynaphthyl)isoxazoles I (R = Me, Et, H; R1 = Me, Et) were obtained by the reaction of naphthopyranones II with HONH2. A mixture of II (R = R1 = Me), HONH2.HCl, and pyridine in EtOH was refluxed 24 h to give I (R = R1 = Me).

IT 82776-43-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 82776-43-0 CAPLUS
CN 1H-Naphtho[2,1-b]pyran-1-one, 3-(diethylamino)-, oxime (9CI) (CA INDEX NAME)

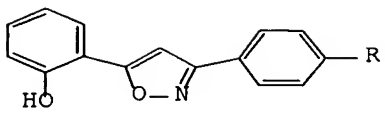


L9 ANSWER 18 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1982:438715 CAPLUS Full-text
DOCUMENT NUMBER: 97:38715
TITLE: Reaction of hydroxylamine with 4'-substituted flavone derivatives
AUTHOR(S): Witczak, Zbigniew; Krolikowska, Maria
CORPORATE SOURCE: Inst. Fundam. Chem. Sci., Sch. Med., Lodz, 90145, Pol.
SOURCE: Polish Journal of Chemistry (1981), 55(4), 763-73
CODEN: PJCHDQ; ISSN: 0137-5083
DOCUMENT TYPE: Journal
LANGUAGE: English

GI



I

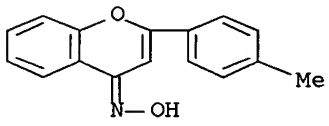


II

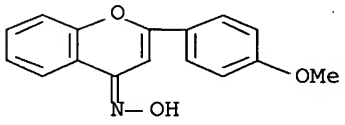
AB Flavones I (X = O, R = OMe, Me, Cl) reacted NH2OH to a give 3:1 mixture of II and I (X = NOH). Similar reaction of I (X = O, R = OH) gave only II (R = OH). Reaction of I (X = O, R = NO2) with NH2OH gave II (R = NO2), 3-(2-hydroxyphenyl)-5-(4-nitrophenyl)isoxazole, and 2- HOC6H4COCH:C(NHOH)C6H4NO2-4.

IT 63645-49-8P 82340-45-2P 82340-46-3P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and acetylation of)

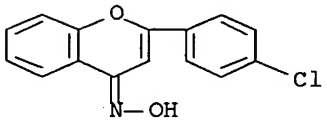
RN 63645-49-8 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methylphenyl)-, oxime (9CI) (CA INDEX NAME)



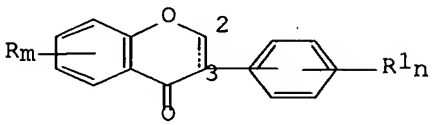
RN 82340-45-2 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-, oxime (9CI) (CA INDEX NAME)



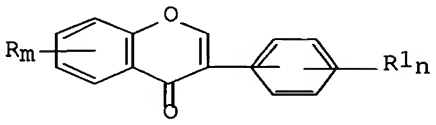
RN 82340-46-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-chlorophenyl)-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 19 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1979:413471 CAPLUS Full-text
DOCUMENT NUMBER: 91:13471
TITLE: Antitumor plants. Part VII. Antineoplastic activity and cytotoxicity of flavones, isoflavones, and flavanones
AUTHOR(S): Edwards, J. Michael; Raffauf, Robert F.; Le Quesne, Philip W.
CORPORATE SOURCE: Sch. Pharm., Univ. Connecticut, Storrs, CT, 06268, USA
SOURCE: Journal of Natural Products (1979), 42(1), 85-91
CODEN: JNPRDF; ISSN: 0163-3864
DOCUMENT TYPE: Journal
LANGUAGE: English
GI



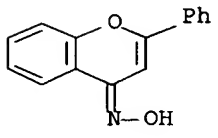
I, 2,3-unsatd.
II, 2,3-satd.



III

AB Two hundred and seventeen natural and synthetic flavonoid derivs. I, II, and III, which were tested in the screening program of the National Cancer Institute, were examd for antineoplastic activity and cytotoxicity. No structure-activity relations were observed Apparently, in spite of occasional activity these compds. do not warrant further investigation as antitumor agents.

IT 22115-89-5
RL: ADV (Adverse effect, including toxicity); BIOL (Biological study)
(antitumor activity and cytotoxicity of, structure in relation to)
RN 22115-89-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 20 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1978:424090 CAPLUS Full-text
DOCUMENT NUMBER: 89:24090
TITLE: Synthesis and pharmacological properties of some

aminoalkyl ethers of heterocyclic ketoximes

AUTHOR(S): Meshcheryakova, L. M.; Orlova, E. K.; Senova, Z. P.; Mochalova, O. A.; Speranskaya, N. P.; Burov, Yu. V.; Zagorevskii, V. A.

CORPORATE SOURCE: Inst. Farmakol., Moscow, USSR

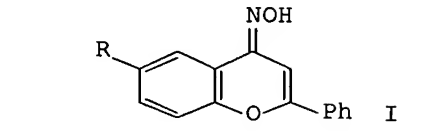
SOURCE: Khimiko-Farmatsevticheskii Zhurnal (1978), 12(4), 50-4
CODEN: KHFZAN; ISSN: 0023-1134

DOCUMENT TYPE: Journal

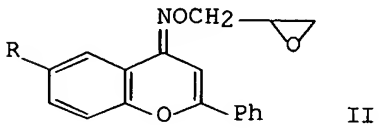
LANGUAGE: Russian

OTHER SOURCE(S): CASREACT 89:24090

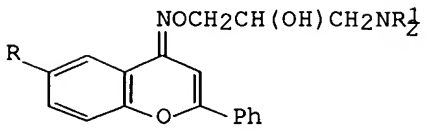
GI



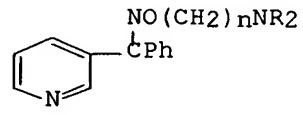
I



II



III



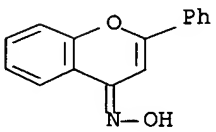
IV

AB Reaction of oximes I (R = Cl, H) with epichlorohydrin gave .apprx.89% II, which when treated with amines gave 30-84% III (NR12 =, NHCHMe2, piperidino, morpholino, 4-methyl-1-piperazinyl). Treatment of the syn and anti isomers of 3-benzoylpyridine oxime with Cl(CH2)nNR2 (n = 2, R = Me, Et; n = 3, R = Me) gave 30-94% IV. All syn- and anti-IV depressed the central nervous system. All IV induced ataxia. IV had analgesic effects at doses close to the LD50. Anti-IV (R = Me, n = 3) had high adrenolytic activity.

IT 22115-89-5 59835-93-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with epichlorohydrin)

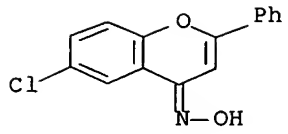
RN 22115-89-5 CAPLUS

CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)

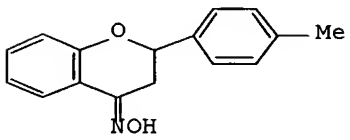


RN 59835-93-7 CAPLUS

CN 4H-1-Benzopyran-4-one, 6-chloro-2-phenyl-, oxime (9CI) (CA INDEX NAME)

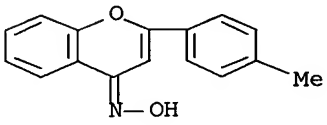


L9 ANSWER 21 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1977:484773 CAPLUS Full-text
DOCUMENT NUMBER: 87:84773
TITLE: Reaction of 2'-hydroxy-4-methylchalcone with hydroxylamine hydrochloride
AUTHOR(S): Krolikowska, Maria; Witczak, Zbigniew
CORPORATE SOURCE: Dep. Org. Chem., Sch. Med., Lodz, Pol.
SOURCE: Roczniki Chemii (1977), 51(3), 611-15
CODEN: ROCHAC; ISSN: 0035-7677
DOCUMENT TYPE: Journal
LANGUAGE: English
GI



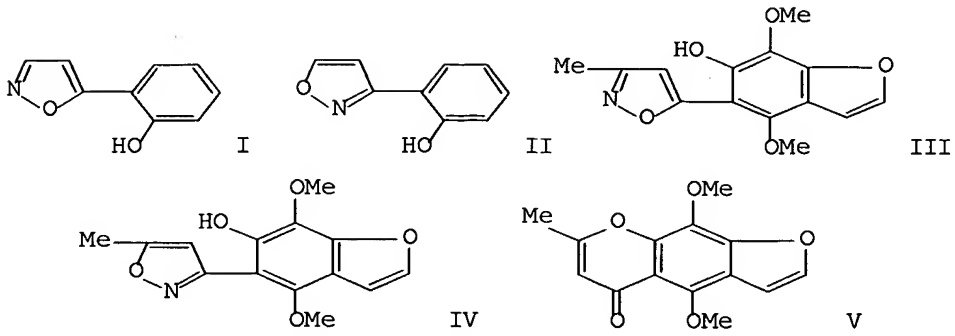
I

AB Reaction of o-HOC6H4COCH:CHC6H4Me-p with NH2OH.HCl yielded 5 compds. depending on reaction conditions; the main product was 4'-methylflavanone oxime (I).
IT 63645-49-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 63645-49-8 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methylphenyl)-, oxime (9CI) (CA INDEX NAME)



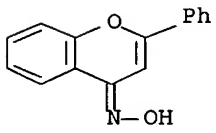
L9 ANSWER 22 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1977:155551 CAPLUS Full-text
DOCUMENT NUMBER: 86:155551
TITLE: Action of hydroxylamine on chromone and khellin. Oxime vs. isoxazoles structures
AUTHOR(S): Beugelmans, Rene; Morin, Christophe

CORPORATE SOURCE: Inst. Chim. Subst. Nat., Gif-sur-Yvette, Fr.
SOURCE: Journal of Organic Chemistry (1977), 42(8), 1356-60
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: English
GI

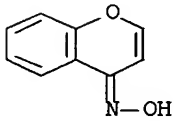


AB Chromone reacted with HONH2 under the usual conditions to give I and II but no oxime. Similarly, III and IV were obtained from khellin (V). When the reactions were performed with H2OH.HCl in anhydrous MeOH, the oximes were obtained. 13C NMR data were given.

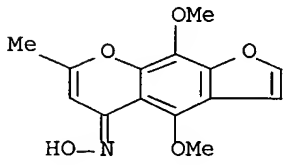
IT 22115-89-5
RL: RCT (Reactant); RACT (Reactant or reagent)
(NMR of carbon-13 in)
RN 22115-89-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)



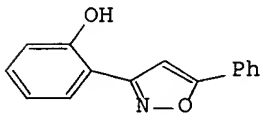
IT 61348-46-7P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(preparation and carbon-13 NMR of)
RN 61348-46-7 CAPLUS
CN 4H-1-Benzopyran-4-one, oxime (9CI) (CA INDEX NAME)



IT 61348-53-6P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 61348-53-6 CAPLUS
CN 5H-Furo[3,2-g][1]benzopyran-5-one, 4,9-dimethoxy-7-methyl-, oxime (9CI) (CA INDEX NAME)

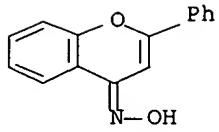


L9 ANSWER 23 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1977:43601 CAPLUS Full-text
DOCUMENT NUMBER: 86:43601
TITLE: Reactions of derivatives of benzo-γ-pyrone with hydroxylamine. Part III
AUTHOR(S): Basinski, Wlodzimierz; Jerzmanowska, Zofia
CORPORATE SOURCE: Inst. Chem., Sch. Med., Lodz, Pol.
SOURCE: Roczniki Chemii (1976), 50(6), 1067-73
CODEN: ROCHAC; ISSN: 0035-7677
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 86:43601
GI

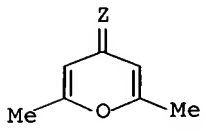
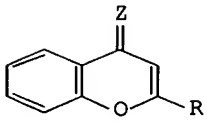
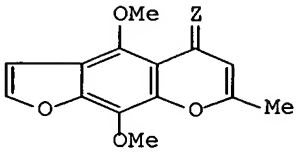


I

AB Reaction of flavone with NH2OH.HCl in C5H5N gave a 3:2 mixture of flavone oxime and the isoxazole I. Mass spectrum of I and the reaction mechanism are discussed.
IT 22115-89-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 22115-89-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)



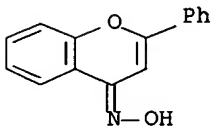
L9 ANSWER 24 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1977:5261 CAPLUS Full-text
DOCUMENT NUMBER: 86:5261
TITLE: Action of reactive nucleophiles (hydroxylamine and hydrazine) on γ-pyrones
AUTHOR(S): Beugelmans, Rene; Morin, Christophe
CORPORATE SOURCE: Inst. Chim. Subst. Nat., CNRS, Gif sur Yvette, Fr.
SOURCE: Tetrahedron Letters (1976), (25), 2145-8
CODEN: TELEAY; ISSN: 0040-4039
DOCUMENT TYPE: Journal
LANGUAGE: French
OTHER SOURCE(S): CASREACT 86:5261
GI


I

II

III

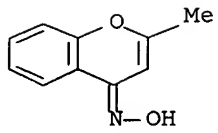
AB Reaction of the γ-pyrones I (Z = O), II (Z = O, R = H, Me, Ph), and III (Z = O) with NH2OH.HCl or NH2NH2.HCl in anhydrous MeOH gave 31-85% oximes I-III (Z = NOH) and 40-70% of the corresponding azines (R = H, Ph), resp. The mechanism for the reaction is discussed and involves formation of 4-hydroxypyrilium ion, which then undergoes attack by base at the 4-position.

IT 22115-89-5P 61169-98-0P 61169-99-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

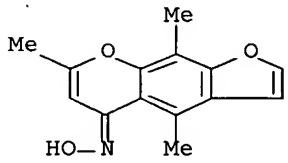
RN 22115-89-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)



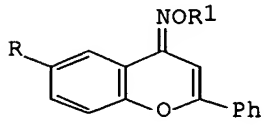
RN 61169-98-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-methyl-, oxime (9CI) (CA INDEX NAME)



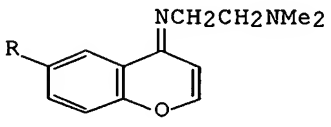
RN 61169-99-1 CAPLUS
CN 5H-Furo[3,2-g][1]benzopyran-5-one, 4,7,9-trimethyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 25 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1976:446315 CAPLUS Full-text
DOCUMENT NUMBER: 85:46315
TITLE: Synthesis and pharmacological activity of some derivatives of 4-imino- and oximinoflavenes
AUTHOR(S): Meshcheryakova, L. M.; Tsikalova, T. S.; Orlova, E. K.; Burov, Yu. V.; Speranskaya, N. P.; Zagorevskii, V. A.
CORPORATE SOURCE: Nauchno-Issled. Inst. Farmakol., Moscow, USSR
SOURCE: Khimiko-Farmatsevticheskii Zhurnal (1976), 10(3), 37-41
CODEN: KHFZAN; ISSN: 0023-1134
DOCUMENT TYPE: Journal
LANGUAGE: Russian
OTHER SOURCE(S): CASREACT 85:46315
GI



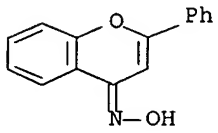
I



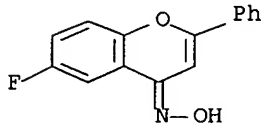
II

AB Flavone O-alkyloximes [I, R = H, Cl, F, R1 = Me, PhCH2, p-O2NC6H4, Me2NCH2CH2, Et2NCH2CH2, Me2N(CH2)3, 4-methyl-1-piperazinylpropyl], useful as sedatives and in treatment of ataxia, were prepared in 40-85% yields by alkylation of the corresponding oximes with R1Cl. II (R = H, Cl) were obtained by treatment of a 4-thioflavone with H2NCH2CH2NMe2.
IT 22115-89-5 59835-92-6 59835-93-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(O-alkylation of)

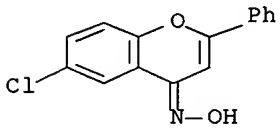
RN 22115-89-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)



RN 59835-92-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-phenyl-, oxime (9CI) (CA INDEX NAME)

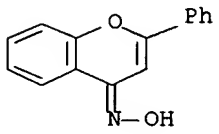


RN 59835-93-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-chloro-2-phenyl-, oxime (9CI) (CA INDEX NAME)

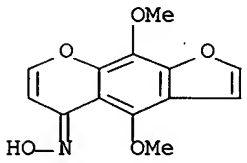


L9 ANSWER 26 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1975:496939 CAPLUS Full-text
DOCUMENT NUMBER: 83:96939
TITLE: Reactions of 4-ethoxyflavylium, chromylium, and furochromylium salts with some amines
AUTHOR(S): Dorofeenko, G. N.; Tkachenko, V. V.; Mezheritskii, V. V.
CORPORATE SOURCE: Rostov. Gos. Univ., Rostov-on-Don, USSR
SOURCE: Khimiya Geterotsiklicheskikh Soedinenii (1975), (4), 465-8
CODEN: KGSSAQ; ISSN: 0132-6244
DOCUMENT TYPE: Journal
LANGUAGE: Russian
OTHER SOURCE(S): CASREACT 83:96939
GI For diagram(s), see printed CA Issue.
AB Amination of I [R = EtO, R1 = H, R2 = Ph (II); R = EtO, R1 = MeO, R2 = H (III)] and IV (R = EtO) by PhNH2 in HOAc gave II-IV (R = PhNH) whereas amination by PhNHNH2 gave II-IV (R = PhNHNH). Condensation of IV (R = EtO) with N2H4 and HONH2 yielded benzofurans V (R3 = 3-pyrazolyl, 3-isoxazolyl), whereas II and HONH2 gave the oxime VI.
IT 22115-89-5P 56430-01-4P
RL: SPN (Synthetic preparation); PREP (Preparation)

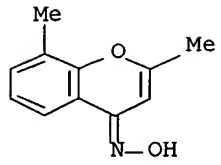
(preparation of)
RN 22115-89-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)



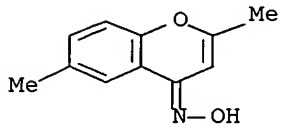
RN 56430-01-4 CAPLUS
CN 5H-Furo[3,2-g][1]benzopyran-5-one, 4,9-dimethoxy-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 27 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1975:479127 CAPLUS Full-text
DOCUMENT NUMBER: 83:79127
TITLE: Reactions of derivatives of benzo-γ-pyrone with hydroxylamine. II
AUTHOR(S): Basinski, Wlodzimierz; Jerzmanowska, Zofia
CORPORATE SOURCE: Inst. Chem., Sch. Med., Lodz, Pol.
SOURCE: Roczniki Chemii (1974), 48(12), 2217-24
CODEN: ROCHAC; ISSN: 0035-7677
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 83:79127
GI For diagram(s), see printed CA Issue.
AB The reaction of 1-(2-hydroxy-3-methylphenyl)-1,3-butanedione with NH2OH gave the dioxime which cyclized in HCl to give I (R = Me, R1 = H) and not 2,8-dimethylchromone oxime (Wittig, G.; Bangert, F., 1925). Similarly, 1-(6-hydroxy-3-methylphenyl)-1,3-butadiene gave I (R = H, R1 = Me) and not 2,6-dimethylchromone oxime.
IT 56686-36-3 56686-37-4
RL: RCT (Reactant); RACT (Reactant or reagent)
(vs. isoxazole derivative, from cyclization of butanedione dioxime derivative)
RN 56686-36-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 2,8-dimethyl-, oxime (9CI) (CA INDEX NAME)



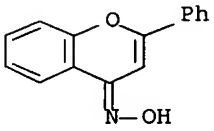
RN 56686-37-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 2,6-dimethyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 28 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1969:114945 CAPLUS Full-text
DOCUMENT NUMBER: 70:114945
TITLE: α-Halo ethers. XL. Flavonoids. 17. Preparation and reactions of some 4,4-dichloroflavene derivatives
AUTHOR(S): Farkas, Istvan; Costisella, Burkhard; Rakosi, Miklos; Gross, Hans; Bognar, Rezso
CORPORATE SOURCE: Univ. Debrecen, Debrecen, Hung.
SOURCE: Chemische Berichte (1969), 102(4), 1333-8
CODEN: CHBEAM; ISSN: 0009-2940
DOCUMENT TYPE: Journal
LANGUAGE: German

AB Treatment of 2-phenyl-7-(R-substituted)-flavones (I, where R = H, AcO, or tetracetyl-β-D-glucopyranosyloxy) with MeOCHCl2 gave 2-phenyl-4,4-dichloro-7-(R-substituted)-2-flavenes (II). The dichloro derivs. of 3-acetoxyflavone, 3-methoxyflavone, and 3,3',4',5,7- pentacetoxyflavone could not be prepared by this method. Treatment of II with AcSH in C6H6 gave 2-phenyl-7-(R-substituted)thioflavones. II (R = H) reacted with MeOH to give I (R = H), with PhSH to give 2-phenyl-4,4-bis(phenylthio)-2-flavene and with R1NH2 to give 2-phenyl-4-(R1N:-substituted)-2-flavene (where R = Ph, C10H21, or OH). 2-Phenylthioflavone reacted similarly to I.

IT 22115-89-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 22115-89-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)



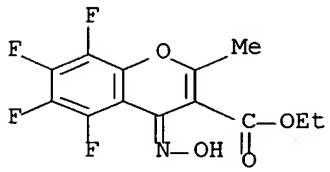
L9 ANSWER 29 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1966:11372 CAPLUS Full-text
DOCUMENT NUMBER: 64:11372
ORIGINAL REFERENCE NO.: 64:2045e-g
TITLE: Synthesis of polyfluorinated chromones and flavones
AUTHOR(S): Vorozhtsov, N. N., Jr.; Barkhash, V. A.; Prudchenko, A. T.; Khomenko, T. I.
CORPORATE SOURCE: Inst. Org. Chem., Novosibirsk
SOURCE: Doklady Akademii Nauk SSSR (1965), 164(5), 1046-9
CODEN: DANKAS; ISSN: 0002-3264
DOCUMENT TYPE: Journal
LANGUAGE: Russian

AB To 0.84 g. Mg shavings was added 1.1 ml. absolute EtOH and 0.1 ml. CCl4 and, after the start of reaction, 3 ml. absolute EtOH in 15 ml. dry C6H6; after dissoln. of Mg 4.14 g. AcCH2CO2Et was added, stirred 1 hr., and the mixture treated with C6F5COCl (6.85 g.), stirred 1 hr., then quenched with aqueous H2SO4 to yield 84.2% 2-methyl-3-carbethoxy-5,6,7,8-tetrafluorochromone, m. 91-1.5°; oxime m. 122-4°. The chromone and PhCH2NH2 in C6H6 in 1 day gave yellow 2-methyl-5,6,7,8-tetrafluorochromone-3-carboxylic acid N-benzylamide, m. 131-6°. Reaction sequence as above but with BzCH2-CO2Et gave 91.5% 3-carbethoxy-5,6,7,8-tetrafluoroflavone, m. 123-4.5°; similar reaction but with 2,3,4,5,6-pentafluorobenzoyl acetate gave 3-carbethoxy-2',3',4',5,5',6,6',7,8-nonafluoroflavone, m. 100-3°. Ir and uv spectra of the products were reported.

IT 4487-62-1P, 4H-1-Benzopyran-3-carboxylic acid, 5,6,7,8-tetrafluoro-2-methyl-4-oxo-, ethyl ester, oxime
RL: PREP (Preparation)
(preparation of)

RN 4487-62-1 CAPLUS

CN 4H-1-Benzopyran-3-carboxylic acid, 5,6,7,8-tetrafluoro-2-methyl-4-oxo-, ethyl ester, oxime (7CI, 8CI) (CA INDEX NAME)



L9 ANSWER 30 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1966:11369 CAPLUS Full-text
DOCUMENT NUMBER: 64:11369
ORIGINAL REFERENCE NO.: 64:2045b-c
TITLE: Polyfluoro derivatives of γ-benzopyrone
AUTHOR(S): Vorozhtsov, N. N., Jr.; Barkhash, V. A.; Prudchenko, A. T.; Khomenko, T. I.
CORPORATE SOURCE: Inst. Org. Chem., Novosibirsk
SOURCE: Zhurnal Obshchei Khimii (1965), 35(8), 1501-2
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Russian

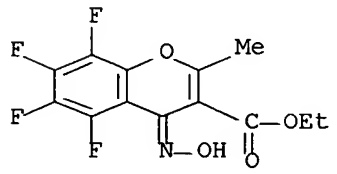
AB Condensation of C6F5COCl with EtOMg derivative of Et acetoacetate in C6H6 (1 hr. at 20° and 15 min. at 50°) gave 84.2% 2-methyl-3-carbethoxy-5,6,7,8-

tetrafluorochromone, m. 91-1.5°; oxime m. 122-4°. Similar reaction with EtOMg derivative of Et benzoylacetate gave 91.5% 3-carbethoxy-5,6,7,8-tetrafluoroflavone, m. 123-4.5°, while condensation with the EtOMg derivative of Et pentafluorobenzoylacetate gave 75.3% 3-carbethoxy-2',3',4',5,5',6,6',7,8- nonafluoroflavone, m. 100-3°. The cyclization probably took place by elimination of the o-F atom.

IT 4487-62-1P, 4H-1-Benzopyran-3-carboxylic acid, 5,6,7,8-tetrafluoro-2-methyl-4-oxo-, ethyl ester, oxime
RL: PREP (Preparation)
(preparation of)

RN 4487-62-1 CAPLUS

CN 4H-1-Benzopyran-3-carboxylic acid, 5,6,7,8-tetrafluoro-2-methyl-4-oxo-, ethyl ester, oxime (7CI, 8CI) (CA INDEX NAME)



L9 ANSWER 31 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1965:9023 CAPLUS Full-text

DOCUMENT NUMBER: 62:9023

ORIGINAL REFERENCE NO.: 62:1626c-e

TITLE: Cupressuflavone, a new member of the biflavonyl group

AUTHOR(S): S. Murti, V. V.; Raman, P. V.; Seshadri, T. R.

CORPORATE SOURCE: Univ. Delhi, India

SOURCE: Tetrahedron Letters (1964), (39-40), 2995-7
CODEN: TELEAY; ISSN: 0040-4039

DOCUMENT TYPE: Journal

LANGUAGE: English

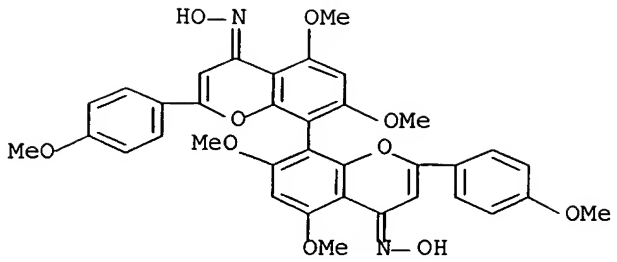
GI For diagram(s), see printed CA Issue.

AB Air-dried leaves of Cupressus torulosa (or of C. sempervirens) exhaustively extracted with hot Me2CO, the concentrate extracted with hot ligroine to remove waxes and chlorophyll, the residue refluxed in alc. and filtered, and the residual greenish-yellow solid extracted with hot Me2CO yielded yellow solid cupressuflavone (I), m. above 360° (MeOH-C5H5N), containing no MeO or C-Me groups; hexaacetate m. 251-3°; tetra-Me ether m. 259-61°; hexa-Me ether m. 295-7°; hexa-Et ether m. 267-9° (oxime m. 290-1°). Degradation of the hexa-Me ether with absolute alc. alkali or alkaline H2O2 gave p-MeOC6H4CO2H as the only recognizable product. The N.M.R. spectrum of the Me ether in CDCl3 was consistent with a sym. dimeric structure. Since the hexa-Me ether was found to be identical with 8,8''-biapigeninyl hexamethyl ether synthesized by Nakazawa (CA 59, 1574a) it was concluded that I is the previously unknown 8,8''-biapigeninyl.

IT 1065-78-7P, Cupressuflavone, hexamethyl ether, dioxime
RL: PREP (Preparation)
(preparation of)

RN 1065-78-7 CAPLUS

CN 8,8''-Biflavone, 4',4''',5,5'',7,7''-hexamethoxy-, dioxime (7CI, 8CI) (CA INDEX NAME)



L9 ANSWER 32 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1963:475197 CAPLUS Full-text
DOCUMENT NUMBER: 59:75197
ORIGINAL REFERENCE NO.: 59:13932d-h,13933a-b
TITLE: Linear benzochromanones and benzochromones. II. Synthesis of 6,7-cyclohexenochromanone, 6,7-benzochromanone, 6,7-cyclohexenochromone, and 6,7-benzochromone
AUTHOR(S): Bell, K. H.; Duewell, H.
CORPORATE SOURCE: Univ. New South Wales, Tighe's Hill
SOURCE: Australian Journal of Chemistry (1963), 16(4), 690-4
CODEN: AJCHAS; ISSN: 0004-9425
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.

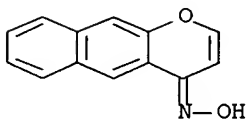
AB cf. CA 59, 5113h. The reactions described previously were extended to the preparation of 6,7benzochromanone (I). Thus, a mixture of 72 g. β-C10H7OH, 250 ml. EtOH, and 1 ml. glacial AcOH was hydrogenated in a rocking autoclave at 150-60° and an initial pressure of 120 atmospheric, using 2 g. W-4 Raney Ni. After 30 min., the catalyst was filtered off, the filtrate evaporated in vacuo, and Et2O added. The solution was extracted with 2% NaOH, which exts. were combined, acidified, and extracted with Et2O. The organic extract was dried, evaporated, and distilled to give 49% 5,6,7,8-tetrahydro-2- naphthol (II), b3 133-4°, m. 59-60° (petr. ether). A mixture of 14.8 g. II, 27 ml. CH2: CHCN, and 1 ml. Triton B (40%) was refluxed 24 hrs. on a H2O bath, cooled, poured into 500 ml. cold 2% NaOH, and extracted with Et2O. The organic extract was washed (2% NaOH, H2O), dried, evaporated, and distilled to give 60% 3-(5,6,7,8-tetrahydro-2-naphthyloxy)propionitrile (III), b2 173-5°, m. 45° (petr. ether). A mixture of 20 g. III, 1200 ml. concentrated HCl, and 600 ml. H2O was refluxed 24 hrs., cooled, and filtered to give 83% 3-(5,6,7,8-tetrahydro-2-naphthyloxy)propionic acid (IV), m. 157.5-8.0° (petr. ether). A mixture of 10 g. IV and polyphosphorie acid (prepared by heating 200 g. P2O5 and 150 ml. 85% H3PO4 at 130-40° for 2-3 hrs.) was heated 1 hr. at 100-5° with occasional shaking, cooled, poured into ice H2O, and extracted with C6H6. The organic extract was washed, dried, evaporated, and distilled to give 67% 6,7-cyclohexenochromanone (V), b2 155-6°; 2,4- dinitrophenylhydrazone m. 269-70° (CHCl3-MeOH); oxime m. 122.5-3.0° (petr. ether). A mixture of 2 g. V and 0.2 g. 30% Pd-C was heated 1 hr. on a metal bath at 230-40° under a stream of CO2, cooled, Et2O added, filtered, and evaporated The residue was chromatographed on 200 g. Al2O3, elution of which with petr. ether gave 0.8 g. putative 6,7-benzochroman (VI), m. 110-11° (MeOH). Elution with 1:1 C6H6-pert. ether gave 0.25 g. I, m. 131-2° (aqueous MeOH); 2,4-dinitrophenylhydrazone m. 252-4° (decomposition) (CHCl3-MeOH). To avoid reduction, a mixture of 1.2 g. V and 0.38 g. S was heated on a metal bath to 250° under a stream of CO2, kept 10 min. at 250°, and worked up as before to give 24% I. In an alternate

approach, a solution of 30 g. II in 750 ml. 2% NaOH was treated successively with 300 g. crushed ice and 45 ml. Ac2O, shaken vigorously 20 min., and extracted with CCl4. The organic extract was washed (N Na2CO3, H2O), dried, evaporated, and distilled to give 78% 5,6,7,8-tetrahydro-2-naphthyl acetate (VII), b3 137-8°. VII was converted in 48% yield by the method of O'Farrell, et al. (CA 50, 9356d) to 5,6,7,8-tetrahydro-2-hydroxy-3-acetonaphthone (VIII), m. 71-2° (MeOH). A solution of 5 g. VIII and 10 ml. HCO2Et (dried over K2CO3 and freshly distilled) in 25 ml. absolute Et2O was added with shaking and cooling to 2 g. powdered Na and 25 ml. absolute Et2O, left 24 hrs. at room temperature, and decomposed carefully with ice H2O. The separated aqueous layer was extracted with Et2O and acidified with dilute H2SO4 to precipitate 72% 5,6,7,8-tetrahydro-2- hydroxy-3-naphthoylacetaldehyde (IX), m. 130-1° (C6H6). A mixture of 1 g. IX, 25 ml. EtOH, and 5 ml. concentrated H2SO4 was refluxed 1 hr., cooled, poured into ice H2O, and filtered to give 87% cyclohexenochromone (X), m. 123° (aqueous EtOH); oxime m. 166-7° (C6H6). VIII was converted by the method of O'Farrell, et al. (loc. cir.), in 46% yield to 2-hydroxy-3-acetonaphthone (XI), m. 112-13° (petr. ether). XI was converted by the method used for IX in 70% yield to 2-hydroxy-3- naphthoylacetaldehyde (XII), m. 238-40° (decomposition) (C6H6). Cyclization of XII was carried out as described for IX to give 84% 6,7-benzochromone (XIII), m. 137-8° (aqueous EtOH); oxime m. 189-91° (decomposition) (aqueous EtOH). A mixture of 20 mg. X, 1 ml. glacial AcOH, and 2.8 mg. PtO2 was shaken under H at room temperature and pressure until uptake was complete, filtered, and evaporated in vacuo. The residual oil was converted directly to the 2,4-dinitrophenylhydrazone of V. Similarly, XIII was reduced to I, identified as its 2,4-dinitrophenylhydrazone. Ultraviolet maximum and min. are reported for the title compds.

IT 96200-32-7P, 4H-Naphtho[2,3-b]pyran-4-one, oxime
96679-42-4P, 4H-Naphtho[2,3-b]pyran-4-one, 6,7,8,9-tetrahydro-, oxime
RL: PREP (Preparation)
(preparation of)

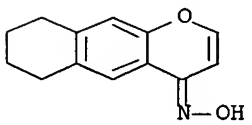
RN 96200-32-7 CAPLUS

CN 4H-Naphtho[2,3-b]pyran-4-one, oxime (7CI) (CA INDEX NAME)



RN 96679-42-4 CAPLUS

CN 4H-Naphtho[2,3-b]pyran-4-one, 6,7,8,9-tetrahydro-, oxime (7CI) (CA INDEX NAME)



L9 ANSWER 33 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1963:408822 CAPLUS Full-text
DOCUMENT NUMBER: 59:8822
ORIGINAL REFERENCE NO.: 59:1574d-h,1575a-f
TITLE: Syntheses of nuclear-substituted flavonoids and allied compounds. IX. Syntheses of the tetramethyl ether and dimethyl ether of ginkgetin
AUTHOR(S): Nakazawa, Koichi
CORPORATE SOURCE: Coll. Pharm., Gifu, Japan
SOURCE: Chemical & Pharmaceutical Bulletin (1962), 10, 1032-8
CODEN: CPBTAL; ISSN: 0009-2363
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.

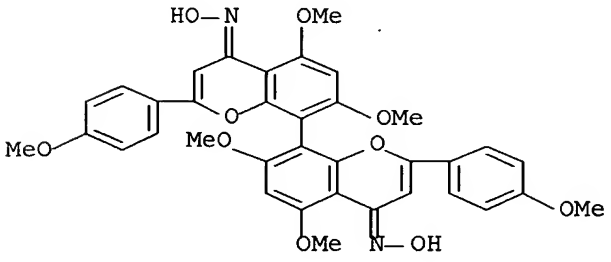
AB cf. CA 51, 3579h. The structure of ginkgetin as I was confirmed by synthesis of its di-Me and tetra-Me ethers. A mixture of 152.1 g. anisic acid, 253.8 g. iodine powder, 150 g. H_2SO_4, and 800 cc. AcOH was stirred 90 min. at 40-50° with addition of 100 g. concentrated HNO_3 and 150 cc. AcOH. After warming at 50° 30 min., the mixture was diluted with 1 l. H_2O to give 203 g. 3-iodoanisic acid, m. 238°. This (139 g.) was added to a mixture of 114 g. PCl_5 and 100 cc. $CHCl_3$, warmed on a steam bath till evolution of HCl ceased and then distilled to give 138 g. 3-iodoanisyl chloride, b12-13 183-5° (solidified). A mixture of 49 g. phloracetophenone 2,4-di-Me ether, prepared by methylation of phloracetophenone with Me_2SO_4 and K_2CO_3 in Me_2CO, 89 g. 3-iodoanisyl chloride, and 120 cc. pyridine was heated at 110° 10 min., cooled, and treated with 150 cc. MeOH to give 86.6 g. 2-acetyl-3,5-dimethoxyphenyl 3-iodoanisate, m. 158°. Heating a mixture of 45.6 g. of this ester, 14 g. KOH powder, and 100 cc. pyridine on a steam bath 5 min. gave a yellow K compound which was decomposed with 50 cc. AcOH and the mixture treated with 100 cc. MeOH to give 18.2 g. 1-(3-iodo-4-methoxyphenyl) - 3 - (2 - hydroxy - 4,6-dimethoxyphenyl)-1,3-propanedione, m. 168°. A hot solution of 45.6 g. of the diketone in 600 cc. AcOH was mixed with 100 g. 20% H_2SO_4 in AcOH and after 10 min. poured into 2 l. H_2O to give 39.8 g. 3'-iodo-4',5,7-trimethoxyflavone (II), m. 223°. For the other part of the mol. was synthesized 8-iodo-4',5,7-trimethoxyflavone (III). A solution of 127 g. iodine in 150 cc. Me_2CO was mixed with a solution of 98.1 g. phloracetophenone 2,4-di-Me ether in 650 cc. AcOH, cooled in an ice bath, and treated with 50 g. concentrated HNO_3 in 60 cc. AcOH during 40 min. to give a precipitate of 120.8 g. 2-hydroxy-3-iodo-4,6 dimethoxyacetophenone (IV), m. 201°. The structure of IV was proved by its conversion into benzyl 2-iodo-3,5-dimethoxyphenyl ether (V) which was synthesized by two unambiguous syntheses. A mixture of 16.1 g. IV, 7 g. $PhCH_2Cl$, 15 g. K_2CO_3, and 35 cc. $HCONMe_2$ was refluxed in an oil bath 1 hr., cooled, and treated with H_2O to give 16.5 g. 2-benzyloxy-3-iodo-4,6-dimethoxyacetophenone (Va), m. 136°. A mixture of 4.1 g. of Va, 22.3 cc. 10% NaClO, and 11 cc. pyridine was heated at 60° 1 hr., diluted with H_2O, filtered and the filtrate acidified to give 2.2 g. 2-benzyloxy-3-iodo-4,6- dimethoxybenzoic acid, m. 183° (decomposition). When 2.1 g. of this acid was heated near its m.p. 1 min. it gave CO_2 and 0.45 g. V, m. 88°. Longer heating resulted in loss of iodine and resinification. In the 1st synthesis of V, 10.6 g. Me 2-hydroxy-4,6-dimethoxybenzoate was benzoylated as above to give an oily residue which was boiled with 200 g. 20% EtOH-KOH 15 min. and acidified with HCl to give 8.2 g. 2-benzyloxy-4,6-dimethoxybenzoic acid, m. 164° (decomposition). The Ag salt of this acid was prepared by adding 10 cc. N $AgNO_3$ to a solution of 2.9 g. acid in 100 cc. 0.1N NaOH to give 3.5 g. of the Ag salt. To a mixture of 2 g. Ag salt and 30 cc. CCl_4 at 85-90° was gradually added 1.3 g. iodine to give 0.7 g. V, m. 88°, identical with the above sample. In the 2nd synthesis of V, 10.6 g. Me 4-hydroxy-2,6-dimethoxybenzoate was benzylated to give 13.5 g. Me 4-benzyloxy-2,6-dimethoxybenzoate, m. 99°. It was iodinated by treating a mixture of 6 g. of the ester and 5.1 g. iodine in 50 cc. AcOH with 2 g. HNO_3 at 50°. After 2 hrs., it gave Me 3-iodo-4-benzyloxy-2,6-dimethoxybenzoate (Vb), m. 118°. On hydrolysis of 4.3 g. of this ester by boiling with 50 g. 20% EtOH-KOH 15 min.

and acidification with HCl Vb gave 3-iodo-4-benzyloxy-2,6- dimethoxybenzoic acid, m. 169° (decomposition), 2.1 g. of which on decarboxylation gave 1.3 g.V. The synthesis of the isomeric substance benzyl 4-iodo-3,5-dimethoxyphenyl ether (VI) was also carried out. Me 2,6-dimethoxy-4-benzyloxybenzoate (6 g.) prepared as above was hydrolyzed to 4.1 g. 2,6-dimethoxy-4-benzyloxybenzoic acid, m. 172° (decomposition) which was converted into 2.8 g. of its Ag salt. Iodination of 2 g. Ag salt as above gave 0.9 g. VI, m. 120°. For the synthesis of III, a mixture of 80.5 g. IV, 51.2 g. anisoyl chloride, and 120 cc. pyridine was treated as in the synthesis of II to give 80.9 g. 2-acetyl-6-iodo-3,5- dimethoxyphenyl anisate, m. 181°. Similarly, a mixture of 45.6 g. of this ester, 11.2 g. KOH, and 100 cc. pyridine gave 35.6 g. 1-(p-methoxyphenyl)-3-(2-hydroxy-3-iodo-4,6-dimethoxyphenyl)-1,3- propanedione. The cyclization of 45.6 g. of this diketone in 500 cc. AcOH with 100 g. 20% H_2SO_4 in AcOH gave 37.2 g. III, m. 239-40°. For the synthesis of biflavone, a mixture of 4.4 g. II, an equal amount of III, 8.8 g. Cu powder, and 30 cc. HCONMe2 was refluxed 4 hrs., filtered hot, and washed with 10 cc. EtOH. The filtrate was diluted with 200 cc. EtOH and left overnight to give 1.5 g. I tetra-Me ether, m. 238°, identical in m.p., mixed m.p., and infrared spectrum with the tetra-Me ether of natural I. The dioxime of the synthetic product prepared by boiling with $NH_2OH.HCl$ and AcOK in pyridine m. 252° and was identical with that of the natural material. A solution of 1.32 g. of the synthetic I tetra-Me ether and 1.32 g. $AlCl_3$ in 10 cc. $PhNO_2$ was heated at 110° 1 hr. to give I di-Me ether, m. 282°, identical in all respects with the di-Me ether of natural I. For comparison, the biflavone compds. from 2 mols. of II and from two mols. of III were also prepared A mixture of 2.2 g. II and 2.2 g. Cu powder in 8 cc. HCONMe2 treated as above gave 10 mg. 3',3'''-biflavone compound VII, m. 354°. Similarly, 2.2 g. III gave 0.54 g. 8,8''-biflavone compound VIII, m. 297°; dioxime m. 294°.

IT 1065-78-7P, 8,8''-Biflavone, 4',4''',5,5'',7,7''-hexamethoxy-, dioxime 107225-53-6P, 3''',8-Biflavone, 4',4''',5,5'',7,7''-hexamethoxy-, dioxime
RL: PREP (Preparation)
(preparation of)

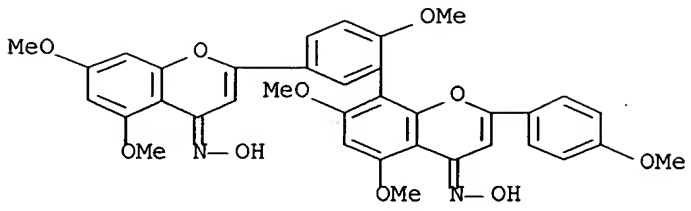
RN 1065-78-7 CAPLUS

CN 8,8''-Biflavone, 4',4''',5,5'',7,7''-hexamethoxy-, dioxime (7CI, 8CI) (CA INDEX NAME)



RN 107225-53-6 CAPLUS

CN 3''',8-Biflavone, 4',4''',5,5'',7,7''-hexamethoxy-, dioxime (7CI) (CA INDEX NAME)



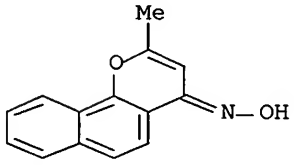
L9 ANSWER 34 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1963:59643 CAPLUS Full-text
DOCUMENT NUMBER: 58:59643
ORIGINAL REFERENCE NO.: 58:10160f-h,10161a-f
TITLE: 4-Pyrones. II. Action of alkylamines on 2-methyl- and 2-styrylnaphtho[1,2-b]-4-pyrones and on 2,6-distyryl-4-pyrones and their sulfur analogs
AUTHOR(S): Elkaschef, Mohamed A. F.; Nosseir, Michael H.; Kader, Awatef Abdel
CORPORATE SOURCE: Natl. Res. Centre, Cairo
SOURCE: Journal of the Chemical Society (1963) 440-4
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 58:59643
GI For diagram(s), see printed CA Issue.
AB cf. CA 55, 2634f. Ia (R = Me, X = O) (I) (1 g.) and 33% aqueous MeNH2 (II) (10 cc.) in EtOH (5C cc.) refluxed 8 hrs. gave on cooling 2-(3-methylamino-2-butenoyl)-1-naphthol (0.5 g.), m. 163°, yellow crystals (EtOH). Similarly, BuNH2 and PhCH2NH2 gave analogous products. Ia (R = Me, X = S) (III) (0.5 g.) in 50 cc. EtOH refluxed for 8 hrs. with 10 cc. II gave, on evaporation, IIIa (R = Me) (IV) (0.3 g.), m. 142° (petr. ether, b. 70-80°). Similarly, 33% aqueous EtNH2 (10 cc.) gave IIIa (R = Et) (V) (0.4 g.), m. 126°, and 3 cc. BuNH2 gave IIIa (R = Bu) (VI) (0.3 g.), m. 106°. However, PhCH2NH2 and III in EtOH or C6H6, upon refluxing, gave Ia (R = Me, X = NCH2Ph) (VII) (0.24 g.), m. 217°. Hydrolysis of IV, V, or VI (0.5 g.) with 25 cc. 17% HCl for 0.5 hr. gave 2-acetyl-1-naphtho (VIII) (0.3 g.), m. 103°. VII did not undergo this acid hydrolysis and was also recovered unchanged after boiling with II in EtOH for 6 hrs. Refluxing I or III in aqueous ethanolic 20% NaOH (25 cc. 4 hrs., cooling, and acidifying with dilute HCl gave VIII. VIII (0.5 g.) heated 6 hrs. in 25 cc. EtOH with II or EtNH2 (10 cc.), or 3 cc. BuNH2 gave, upon evaporation and recrystn. from petr. ether, b 70-80°, quant. yields of IV, V, and VI, resp. Similarly, Ph CH2NH2 gave IIIa (R = PhCH2), m. 147° (EtOH). A mixture o 3 g. I in 20 cc. EtOH, NaOEt (from 0.4 g. Na), and p-ClC6H4 CHO (IX) (2.4 g.) in 20 cc. EtOH, after standing at room temperature for 2 days, gave Ia (R = p-ClC6H4CH:CH, X = O) (X), yellow crystals, m. 198° (C6H6-EtOH). III (1 g.) and 1 g. IX in 20 cc. absolute EtOH containing 4 drops of piperidine gave, after refluxing 6 hrs., Ia (R = p-ClC6H4CH:CH, X = S) (XI) (0.8 g.), violet crystals, m. 258° (decomposition) (C2H6). In a similar manner, Ia (R = p-MeC6H4CH:CH, X = S) (XII) was prepared, m. 235° (C6H6). In addition, condensation of I or III with the appropriate aldehydes produced Ia (R = PhCH:CH, X = O) (XIII), Ia (R = PhCH:CH, X = S) (XIV), Ia (R = p-MeC6H4CH:CH, X = O) (XV), Ia (R = p-MeOC6H4CH:CH, X = O) (XVI), and Ia (R = p-MeOC6H4CH:CH, X = S) (XVII). Reaction of each of the thiopyrones III, XI, XII, XIV, or XVII (0.5 g.) in 50 cc. EtOH with 0.7 g. HONH2.HCl and 0.7 g. NaOAc in 1 cc. H2O gave, after refluxing 5 hrs., cooling, and diluting with H2O, the following yellow oximes, recrystd. from EtOH (parent, g. yield, m.p. given): III (or I),

0.3, 182° (was unchanged after boiling with II 6 hrs.); XI (or X), 0.35, 244°; XII (or XV), 0.3, 256°; XIV (or XIII), 0.4, 210°; XVII (or XVI); 0.25, 230°. Refluxing each of the pyrones (X, XIII, XV, XVI) or thiopyrones (XI, XIV, XII, XVII) (1 g.) for 8 hrs. in EtOH with II or EtNH2 (10 cc.) or PhCH2NH2 (5 cc.) gave, from C6H6-EtOH, the following XVIIa (R, R', and m.p. given): H, Me (0.6 g. from XIII, 0.9 g. from XIV), 210° (decomposition); Cl, Me (0.9 g. from X or XI), 215° (decomposition); MeO, Me (no reaction with XVI, 1 g. from XVII), 205° (decomposition); H, Et (0.6 g. from XIII, 1 g. from XIV), 187°; Cl, Et (1 g. from X, 0.6 g. from XI), 234°; OMe, Et (no reaction with XVI, 1 g. from XVII), 236°; H, PhCH2 (0.6 g. from XIII, 1 g. from XIV), m. 224° (decomposition); Cl, PhCH2 (0.8 g. from X, 1 g. from XI), 212° (decomposition); OMe, PhCH2 (no reaction with XVI, I g. from XVII), 216°. In a similar manner, XIII treated with 3 cc. BuNH2 gave XVIIa (R = H, R' = Bu) (0.58 g.), m. 188°, and X gave XVIIa (R = Cl, R' = Bu) (0.9 g.), m. 216°. 2,6-Dimethyl-4-pyrone (XVIII) (1 g.) was dissolved in a small quantity of absolute EtOH and treated at room temperature with alc. NaOEt (2 equivs.) and BzH (2 equivs.). The solution was kept for 48 hrs. at room temperature and the product, which usually separated, was dissolved in EtOH. Acidification with dilute HCl precipitated XIX (R = H, X = O) (1 g.), m. 168° (aqueous EtOH). This product did not react with alkylamines. In a similar manner, 2,6-dimethyl-4-thiopyrone (XX) and BzH produced XIX (R = H, X = S) (1 g.), m. 244° (C6H6-EtOH). Similar condensation of XVIII or XX with p-substituted benzaldehydes produced the XIX (R, X, and m.p. given): MeO, O, 199° (EtOH); MeO, S, 191° (C6H6EtOH); Me, O, 192° (EtOH); Me, S, 217° (C6H6); Cl, O, 238° (C6H6-EtOH). These compds. were unchanged on treatment with alkylamines.

IT 92163-42-3P, 4H-Naphtho[1,2-b]pyran-4-one, 2-methyl-, oxime
94872-93-2P, 4H-Naphtho[1,2-b]pyran-4-one, 2-(p-chlorostyryl)-, oxime 94878-46-3P, 4H-Naphtho[1,2-b]pyran-4-one, 2-styryl-, oxime 97015-52-6P, 4H-Naphtho[1,2-b]pyran-4-one, 2-(p-methylstyryl)-, oxime 97015-63-9P, 4H-Naphtho[1,2-b]pyran-4-one, 2-(p-methoxystyryl)-, oxime
RL: PREP (Preparation)
(preparation of)

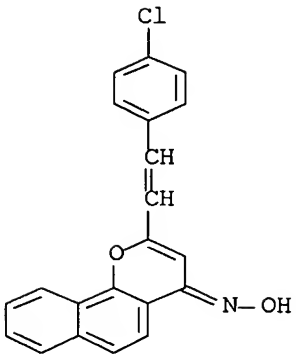
RN 92163-42-3 CAPLUS

CN 4H-Naphtho[1,2-b]pyran-4-one, 2-methyl-, oxime (7CI) (CA INDEX NAME)

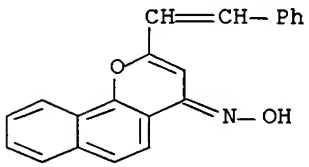


RN 94872-93-2 CAPLUS

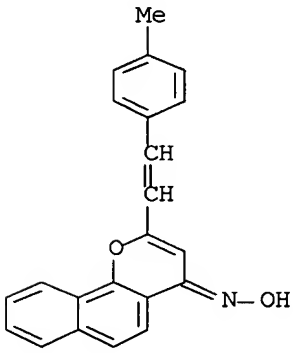
CN 4H-Naphtho[1,2-b]pyran-4-one, 2-(p-chlorostyryl)-, oxime (7CI) (CA INDEX NAME)



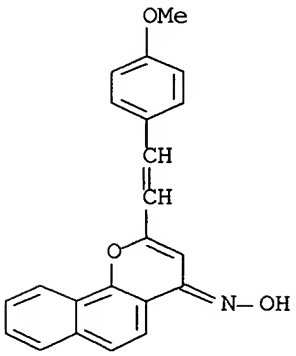
RN 94878-46-3 CAPLUS
CN 4H-Naphtho[1,2-b]pyran-4-one, 2-styryl-, oxime (7CI) (CA INDEX NAME)



RN 97015-52-6 CAPLUS
CN 4H-Naphtho[1,2-b]pyran-4-one, 2-(p-methylstyryl)-, oxime (7CI) (CA INDEX NAME)



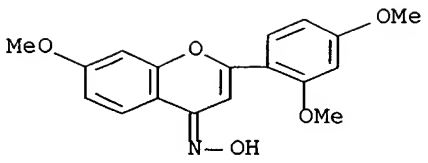
RN 97015-63-9 CAPLUS
CN 4H-Naphtho[1,2-b]pyran-4-one, 2-(p-methoxystyryl)-, oxime (7CI) (CA INDEX NAME)



L9 ANSWER 35 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1960:128929 CAPLUS Full-text
DOCUMENT NUMBER: 54:128929
ORIGINAL REFERENCE NO.: 54:24701g-i,24702a-b
TITLE: 7,2',4'-Trimethoxyflavone
AUTHOR(S): Spatz, Sydney M.; Koral, Marvin
CORPORATE SOURCE: Allied Chem. Corp., Buffalo, NY
SOURCE: Journal of Organic Chemistry (1959), 24, 1381-2
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB In an attempt to synthesize 2,2'-dihydroxy-4,4'-dimethoxydibenzoylmethane (I) to obtain the spectral characteristics, the intermediate 2,2',4,4'-tetramethoxydibenzoylmethane (II) was cleaved with HI by way of the transitory 2-hydroxy-2',4,4'-trimethoxydibenzoylmethane (III) to the title compound (IV). Polyphosphoric acid (400 g.) and 41.4 g. m-(MeO)2C6H4 stirred 3 hrs. at 50-3° in 21.6 g. AcOH and the mixture diluted with 1200 ml. ice H2O, extracted with Et2O and the dried (Na2SO4) extract evaporated yielded 86.1% 2,4-(MeO)2C6H3COMe (V), b3 134-7°, m. 40-3°. Me2SO4 (1675 g.) and 612 g. NaOH in 1300 ml. H2O used in the treatment of 154 g. β-resorcylic acid according to Robinson and Venkataraman (CA 23, 2181) yielded 80% 2,4-(MeO)2C6H3CO2H, m. 99-104°, esterified (126 g.) in 277 ml. C6H6 with 127 g. alc. in the presence of 3 ml. 100% H2SO4 to yield 89 g. 2,4-(MeO)2C6H3CO2Et (VI), b3.5 143-7°. Freshly prepared NaNH2 (0.5 mole) freed from NH3 and taken up simultaneously in anhydrous Et2O, cooled (solid CO2) and treated in 10 min. with 45 g. V in 42 ml. Et2O, kept 5 min. and treated in 10 min. with 52.5 g. VI in Et2O, the mixture refluxed overnight and quenched in ice H2O containing HCl, the Et2O layer dried and evaporated and the reddish yellow solid recrystd. yielded 37.2% yellow crystalline II, m. 131-4°, λ 6.04, 6.24 μ (CCl4). P2O5 (1.2 g.) and 5.1 g. 85% H3PO4 treated at 20° with 5.0 g. finely powdered KI and 1.7 g. II in succession and the mixture stirred 30 min. at 105-10°, poured into ice H2O and the precipitate recrystd. from dilute alc. yielded 75% IV, m. 143.5-5.5°, λ 6.11 μ (CCl4), λ 236, 334 mμ (ε 2200, 2450, MeOH); oxime, m. 204-7°. Attempts to demethylate II to I by means of AlCl3 or 48% HBr failed, though the transitory III may have formed and given IV by a 1,3-prototropic shift of a methylene H atom, followed by cyclodehydration.

IT 101734-73-0P, Flavone, 2',4',7-trimethoxy-, oxime
RL: PREP (Preparation)
(preparation of)
RN 101734-73-0 CAPLUS
CN Flavone, 2',4',7-trimethoxy-, oxime (6CI) (CA INDEX NAME)

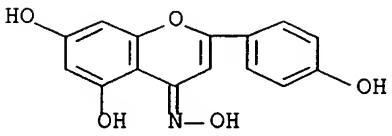


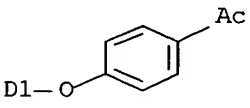
L9 ANSWER 36 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1960:128922 CAPLUS Full-text
DOCUMENT NUMBER: 54:128922
ORIGINAL REFERENCE NO.: 54:24698d-i
TITLE: Chemical constituents of the plants of Coniferae and allied orders. XXXIX. Structure of hinokiflavone, a flavonoid from the leaves of Chamaecyparis obtusa. 3. Structure of substance X and oxoflavone
AUTHOR(S): Kawano, Nobusuke; Fukui, Yoshio
SOURCE: Yakugaku Zasshi (1960), 80, 749-52
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB The phenolic ketone (I) (V in the preceding part) (substance X in title) (100 mg.) in 40 ml. Me2CO, 0.6 ml. Me2SO4 and 1.2 g. K2CO3 was refluxed 10 hrs., the Me2CO removed and the residue with 2% KOH allowed to stand to give 80 mg. I tri-Me ether (II), m. 94-5° (petr. ether). II (50 mg.), 50 mg. NH2OH.HCl, 0.3 ml. C5H5N and 0.3 ml. EtOH refluxed 3 hrs., the solvent removed and the residue treated with dilute AcOH gave II monooxime, m. 156-8° (50% MeOH). II (50 mg.) in 2 ml. C5H5N and 10 ml. 2% KOH, heated on a H2O bath and treated dropwise with 70 mg. KMnO4 in 10 ml. H2O, the solution acidified with HCl and the product extracted with Et2O gave p-[2,4,6-(MeO)3C6H2O]C6H4CO2H (III), m. 191-2°. III (50 mg.) in 4 ml. quinoline and 150 mg. Cu refluxed 4 hrs., the solution with a small amount of Et2O filtered, the filtrate washed with 10% HCl, 2N Na2CO3 and H2O, and concentrated gave 20 mg. 2,4,6-(MeO)3C6H2OPh (IV), m. 94.5° (MeOH). Synthesis of IV: a mixture of 0.54 g. PhOK, 1 g. PhOH, 50 mg. Cu and 1 g. bromophloroglucinol tri-Me ether heated 1 hr. at 180°, the product extracted with C6H6, diluted with Et2O, washed with H2O, 5% NaOH and H2O, the solvent removed, the residue in 2 ml. C6H6 chromatographed through Al2O3, and the 1st effluent concentrated gave 30 mg. phloroglucinol tri-Me ether, m. 55°, and the next effluent gave 100 mg. IV, m. 94.5°. The oxoflavone (V) (IV in the preceding part) (900 mg.) treated with 2.5 ml. Me2SO4 and 30% KOH and the product treated as usual gave 700 mg. tri-O-methyloxoflavone (VI), m. 209-11° (MeOH). V (100 mg.) in 20 ml. Me2CO, 0.4 ml. MeI and 1.5 g. K2CO3 refluxed 2.5 hrs. and the product filtered gave 46 mg. VI; the Me2CO-insol. portion in H2O acidified with HCl and the product filtered gave 10 mg. di-O-methyloxoflavone. VI dioxime m. 144-6°. V monooxime m. 233-4°. V semicarbazone m. 152-4° (decomposition). V (100 mg.) in 1 ml. C5H5N and 0.5ml. Ac2O refluxed 2 hrs. and the product poured in H2O gave tri-O-acetyloxoflavone, m. 205-7° (MeOH). V (1 g.) in 50 ml. 25% KOH refluxed 2 hrs., cooled, the solution made up to 100 ml. with H2O, acidified with H2SO4, the solution filtered and the filtrate kept at 0° gave 200 mg. I, m. 201°; the mother liquor extracted with Et2O, stirred with 5% NaOH, the NaOH layer acidified and the product extracted with Et2O gave p-HOC6H4Ac, m. 108°. Thus, the structure of I was confirmed to be p-[2,4,6-(HO)3C6H2O]C6H4Ac, and that of V must be 8- or 6-(p-acetylphenoxy)-5,7,4-trihydroxyflavone.

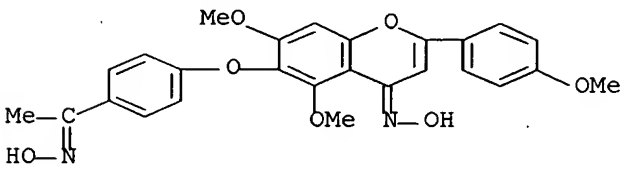
IT 114255-62-8
(Derived from data in the 6th Collective Formula Index (1957-1961))
RN 114255-62-8 CAPLUS
CN Flavone, 6(or 8)-(p-acetylphenoxy)-4',5,7-trihydroxy-, oxime (6CI) (CA

INDEX NAME)





IT 102948-15-2P, Flavone, 6-(p-acetylphenoxy)-4',5,7-trimethoxy-, dioxime
RL: PREP (Preparation)
(preparation of)
RN 102948-15-2 CAPLUS
CN Flavone, 6-(p-acetylphenoxy)-4',5,7-trimethoxy-, dioxime (6CI) (CA INDEX NAME)



L9 ANSWER 37 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1960:128921 CAPLUS Full-text
DOCUMENT NUMBER: 54:128921
ORIGINAL REFERENCE NO.: 54:24698a-d
TITLE: Chemical constituents of the plants of Coniferae and allied orders. XXXVIII. Structure of hinokiflavone, a flavonoid from the leaves of Chamaecyparis obtusa. 2. Composition of hinokiflavone and its degradation in potassium hydroxide solution
AUTHOR(S): Kariyone, Tatsuo; Fukui, Yoshio
SOURCE: Yakugaku Zasshi (1960), 80, 746-9
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

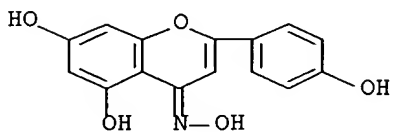
AB cf. CA 54, 3405c. The leaves yielded about 0.1% crude hinokiflavone (I). I (1 g.) treated with 10 ml. C5H5N and 5 ml. Ac2O gave 700 mg. penta-O-acetylhinokiflavone (II), m. 239-40° (decomposition). II (500 mg.) in 50 ml. 2% KOH was refluxed 2 hrs., dilute H2SO4 added, the precipitate taken up in 3 ml. C5H5N and 50 ml. MeOH added to give a pure I, C30H15O10.H2O m. 353-5° (decomposition). I (500 mg.) treated with 5 ml. Me2SO4 and 30% KOH and the product recrystd. (MeOH) gave 400 mg. penta-O-methylhinokiflavone (III), m. 259-60°; oxime, m. 202-3°. Similarly, I, Et2SO4 and 30% KOH gave penta-O-ethylhinokiflavone, m. 249-51°. I (500 mg.) in 2 g. K2CO3, 0.5 ml. MeI and

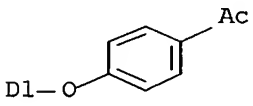
Me2CO refluxed 14 hrs. and the product recrystd. (MeOH) gave 110 mg. tri-O-methylhinokiflavone, m. 259-60°; this, 100 mg., 2 ml. Ac2O and 1 drop H2SO4 gave a diacetate, m. 255°. I (2 g.) in 40 ml. 25% KOH refluxed 1 hr., the solution acidified with dilute H2SO4 and the precipitate filtered gave an oxoflavone (IV), C23H16O7.1.5H2O, m. 256-8° (MeOH), and the mother liquor gave 100 mg. phenolic ketone (V), C14H12O5, m. 201°. The mother liquor extracted with Et2O and 5% NaOH, the NaOH layer acidified and the product extracted with Et2O gave p- hydroxyacetophenone, m. 108-9°.

IT 114255-62-8 124162-05-6
(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 114255-62-8 CAPLUS

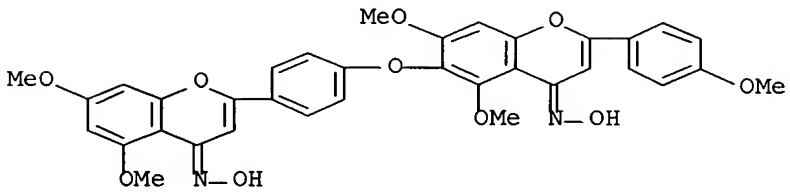
CN Flavone, 6(or 8)-(p-acetylphenoxy)-4',5,7-trihydroxy-, oxime (6CI) (CA INDEX NAME)





RN 124162-05-6 CAPLUS

CN Hinokiflavone, penta-O-methyl-, dioxime (6CI) (CA INDEX NAME)



L9 ANSWER 38 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1960:128920 CAPLUS Full-text

DOCUMENT NUMBER: 54:128920

ORIGINAL REFERENCE NO.: 54:24697g-i,24698a

TITLE: Isoflavones and isoflavanones. V. The alkaline degradation of isoflavanones

AUTHOR(S): Inoue, Naoko

CORPORATE SOURCE: Tohoku Univ., Sendai

SOURCE: Nippon Kagaku Zasshi (1958), 79, 1124-8
CODEN: NPKZAZ; ISSN: 0369-5387

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB cf. ibid. 222; CA 54, 5635c. Isoflavones, such as 7-methoxyisoflavone, 4',7-dimethoxyisoflavone, 5,7-dimethoxyisoflavone (I), and 4',5,7-trimethoxyisoflavone, were heated with 3.0 g. NaOH, 15 cc. H2O, and 25 cc. EtOH and the results obtained compared with those of similar runs with

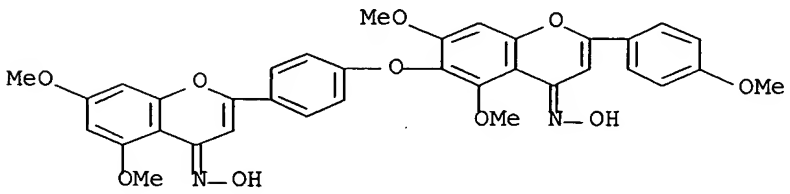
flavanones, such as 7-methoxy-, 4',7-dimethoxy-, 5,7-dimethoxy- (II), and 4',5,7,-trimethoxyisoflavanone. Isoflavones gave HCO2H and phenyl benzyl ketone (III), almost quant., and isoflavanones gave 2.8-7.6% HCHO, 3.1-8.3% III and 3.3-30.4% unchanged original compound II, a new compound was prepared as follows. To 3 g. powdered Na, cooled in ice was added dropwise with stirring 3 g. 2-hydroxy-4,6-dimethoxyphenyl benzyl ketone in 80 cc. HCO2Et, the mixture kept 48 hrs. in an ice box, poured into H2O, HCO2Et evaporated in vacuo, the precipitate filtered off, boiled 1 hr. with 10 cc. AcOH, poured into 300 cc. H2O, and the deposit twice recrystd. from EtOH to yield 2.07 g. I, m. 122-3°. I (1.5 g.) reduced catalytically over 1.0 g. PtO in 80 cc. AcOH gave 1.12 g. II, m. 151°.

IT 124162-05-6

(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 124162-05-6 CAPLUS

CN Hinokiflavone, penta-O-methyl-, dioxime (6CI) (CA INDEX NAME)



L9 ANSWER 39 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1960:128791 CAPLUS Full-text

DOCUMENT NUMBER: 54:128791

ORIGINAL REFERENCE NO.: 54:24625a-i,24626a-i,24627a-c

TITLE: Reaction of propylene oxide, styrene oxide, and cyclohexene oxide with an Ivanov reagent

AUTHOR(S): Blicke, F. F.; Wright, P. E.

CORPORATE SOURCE: Univ. of Michigan, Ann Arbor

SOURCE: Journal of Organic Chemistry (1960), 25, 693-8
CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

AB PhCH2CO2H (I) (272.2 g.) in 1500 ml. C6H6 added dropwise with stirring to iso-PrMgCl (prepared from 112 g. Mg, 361 g. iso-PrCl, and 1 l. Et2O) (the reaction was initiated with 5 ml. EtBr), the mixture refluxed 4 hrs. [preparation of PhCH(MgCl)CO2MgCl (II), an Ivanov reagent], treated dropwise with stirring with 133.6 g. propylene oxide in 5 ml. C6H6, the whole stirred 20 hrs., the Et2O distilled, the residual mixture treated with 400 ml. concentrated HCl in 1.5 l. ice H2O with stirring, the organic layer separated, the aqueous layer extracted with C6H6, the combined C6H6 solns. concentrated to 400 ml., refrigerated 24 hrs., the precipitate filtered off, the filtrate concentrated to 300 ml., cooled 24 hrs., the precipitate filtered off [the filtrate (III) reserved], and the combined ppts. washed with hot petr. ether (b. 60-75°) gave 83.0 g. EtCH(OH)CHPhCO2H (IV), m. 142-3° (PhMe); III stirred with 84 g. NaHCO3 in 1 l. H2O, the aqueous layer separated, extracted with C6H6, the C6H6 solns. combined, concentrated, the residue distilled, the distillate (123 g., b0.3 109°) refrigerated 48 hrs., and the partially solid product filtered off gave 35.3 g. O.CH2.CHMe.CHPh.CO (V), m. 93-4° (petr. ether); the liquid filtrate [83.8 g., presumably a stereoisomer (VI) of V] refluxed with aqueous NaOH until it dissolved, the solution acidified at 0°,

and the separated oil cooled gave V, m. 93-4° (petr. ether), and VI, l iquid, b0.3 109°; the latter refluxed with aqueous NaOH until it dissolved and worked up as above again gave V, m. 93-4°, and VI, liquid, b0.3 109°. EtCHO (116.2 g.) in 500 ml. C6H6 added dropwise to II (from 272.2 g. I) with stirring, the mixture refluxed 6 hrs., hydrolyzed with 333 ml. concentrated HCl in 1.5 l. ice H2O, the layers separated, the aqueous layer extracted with Et2O and C6H6, the combined organic solns. concentrated to smaller volume, the precipitate (167.5 g.) filtered off [the filtrate (VII) reserved], and recrystd. from PhMe gave IV, m. 142-3°; VII concentrated and cooled gave 65.3 g. stereoisomer (VIII) of IV, m. 125-7° (PhMe), neutralization equivalent 196.1. IV (3.8 g.) in 500 ml. (MeOCH2)2 (IX) treated with CH2N2-Et2O, the mixture allowed to stand 2 hrs. at 0°, and the solvents removed in a stream of air gave 3.3 g. Me ester (X) of IV, m. 57-8° (petr. ether). Similarly was prepared 64% Me ester of VIII, b0.3 90°. Na (2.3 g.) dissolved in 250 ml. iso-PrOH, the solution treated with 19.4 g. IV and 17.5 g. Et2NCH2CH2Cl.HCl (XI), the mixture refluxed 16 hrs., filtered, the filtrate concentrated, the residue made alkaline with Na2CO3, extracted with Et2O, the extract dried, evaporated, and the residue recrystd. from petr. ether gave 15.8 g. Et2NCH2CH2 ester of IV, m. 78-80°; di-H citrate m. 96-8° (iso-PrOH). Similarly was prepared 51% Et2NCH2CH2 ester of VIII, b0.5 111°; di-H citrate m. 93-4° (iso-PrOH). X (10.4 g.) in 250 ml. C6H6 added dropwise with stirring to a refluxing mixture of 20 g. Celite, 15 g. P2O5, and 750 ml. C6H6, the mixture stirred and heated 4 hrs., cooled, filtered, the filtrate washed with aqueous NaHCO3, dried, and evaporated gave 9.2 g. oily dehydration product (XII), b20 155°. XII (3.8 g.) dissolved in absolute MeOH, hydrogenated over 0.1 g. PtO2 under an initial pressure of 50 lb. (the calculated amount H was absorbed in 2 hrs.), the mixture filtered, the filtrate evaporated, the residual oil allowed to stand 5 days with 2.2 g. KOH in 25 ml. 90% EtOH, the solution diluted with 150 ml. H2O, extracted with Et2O, cooled, and acidified gave 2.5 g. PrCHPhCO2H (XIII), m. 51-3° (petr. ether). CH2:CHCH2CHPhCO2H (3.5 g.) in 100 ml. absolute MeOH hydrogenated over 0.1 g. PtO2 under an initial pressure of 50 lb., the mixture filtered, the filtrate evaporated, and the residue recrystd. from petr. ether gave XIII, m. 51-3°. VI (17.6 g.) added to 15 g. NaOH in 75 ml. H2O, the mixture heated until a solution was obtained, the solution stirred and kept hot while a hot solution of 43.5 g. MgSO4.7H2O in 33 ml. H2O was slowly added, the mixture cooled to 10°, treated dropwise during 2 hrs. with 5.5 ml. Br, stirred several hrs. in the cold, acidified with 33 ml. 32% H2SO4, the precipitate filtered off, and washed with PhMe gave 6.2 g. HO2CCHPhCHMeCO2H (XIV), m. 169-72° (H2O). V (17.6 g.) treated similarly gave 4.1 g. XIV, m. 169-72°. VI (8.8 g.) shaken 18 hrs. with 15 ml. concentrated aqueous NH3, the precipitate filtered off, the filtrate treated with a stream of air (to remove excess NH3), and the combined ppts. (9.4 g.) recrystd. from EtAc gave HOCH2CHMeCHPrCONH2 (XV), m. 145-6°. V (4.4 g.) in 200 ml. absolute EtOH treated slowly during 6 hrs. with NH3, the EtOH evaporated, and the residue recrystd. from EtAc gave XV, m. 145-6°. XV (11.4 g.) dissolved in 100 ml. anhydrous tetrahydrofuran (XVI), the solution added slowly with stirring to 5.3 g. LiAlH4 in 250 ml. XVI, the mixture stirred 4 hrs., treated dropwise with 10 ml. H2O with stirring, filtered, the filtrate concentrated, and the residue distilled gave 5.1 g. HOCH2CHMeCHPhCH2NH2, b0.7 132°. Styrene oxide (48.0 g.) in 250 ml. C6H6 added dropwise with stirring to II (from 54.4 g. I), the mixture refluxed 72 hrs., hydrolyzed with 67 ml. concentrated HCl in 1 l. ice H2O, the precipitate filtered off, the organic layer partially evaporated (more precipitate separated), and the combined ppts. recrystd. from EtOH gave 41.2 g. PhCH2CH(OH)CHPhCO2H (XVII), m. 201-2°; the organic layer concentrated further and the resulting precipitate recrystd. from PhMe gave 17.2 g. stereoisomer (XVIII) of XVII, m. 176-8°. XVII (128 g.), 1.5 l. MeOH, and 5 ml. concentrated H2SO4 refluxed 20 hrs., the solution concentrated, and the resulting precipitate recrystd. from petr. ether gave 107 g. Me ester (XIX) of XVII, m. 98-9°; XVII with CH2N2 gave XIX, m. 98-9°. XVIII (17.0 g.) in 500 ml. dioxane cooled to 0°, treated with twice the calculated amount of CH2N2 in

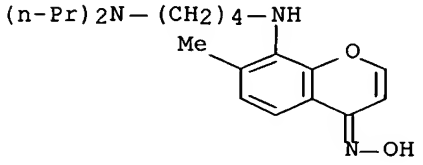
100 ml. Et2O, the solvents removed, and the product recrystd. from petr. ether gave 17.0 g. Me ester (XX) of XVIII, m. 49-50°. To 2.3 g. Na in 250 ml. iso-PrOH was added 25.6 g. XVII followed by 17.5 g. XI, the mixture refluxed 16 hrs., filtered, the filtrate concentrated to small volume, the precipitate (5.0 g.) filtered off [the filtrate (XXI) reserved], and recrystd. from iso-PrOH to give Et2NCH2CH2 ester (XXII) of XVII HCl salt (XXIII), m. 100-1°; XXI made alkaline with aqueous Na2CO3 extracted with Et2O, the extract dried, evaporated, and the residue distilled gave 13.5 g. XXII, b0.7 106; methobromide m. 158-9° (iso-PrOH). Similarly was prepared the Et2NCH2CH2 ester (XXIV) of XVIII HCl salt, m. 100-1°, mixed m.p. (with XX) 85-95°; XXIV methobromide m. 129-30° (iso-PrOH). XIX in 250 ml. C6H6 added dropwise with stirring to a refluxing mixture of 50 g. Celite, 140 g. P2O5, and 1 l. C6H6, the mixture stirred and refluxed 4 hrs., filtered, the filtrate evaporated, and the residue distilled gave 77% dehydration product (Me ester of 2,4-diphenyl-2- or 3-butenoic acid) (XXV), b0.5 151°. XX dehydrated similarly gave 78% XXV, b0.7 150°. PhCH2CHO (18.0 g.) in 75 ml. C6H6 added dropwise with stirring to II (from 20.4 g. I), the mixture refluxed 12 hrs., hydrolyzed with 25 ml. concentrated HCl in 250 ml. H2O, the precipitate filtered off [the filtrate (XXVI) reserved], and recrystd. from EtOH gave 330 g. XVII, m. 204-6°, converted with CH2N2-Et2O in IX to 85% XIX, m. 98-9°; XXVI concentrated and cooled gave 2.5 g. XVIII, m. 172-4° (PhMe), converted with CH2N2-Et2O in IX to 51% XX, m. 54-5°. XVII (25.6 g.) and 50 ml. Ac2O refluxed 8 hrs., the Ac2O removed, and the residue distilled gave 6.5 g. lactone of XVII, b0.4 170°, solidifying and m. 125-6° (Et2O). XVIII (12.8 g.), 250 ml. MeOH, and 1 ml. concentrated H2SO4 refluxed 20 hrs., concentrated, the residue treated with hot Et2O, the mixture filtered, the filtrate cooled, evaporated, and the product recrystd. from Et2O gave 9.2 g. lactone of XVIII, m. 82-4°. XVII (10.2 g.) in 500 ml. Et2O added to 1.5 g. LiAlH4 in 250 ml. Et2O, the mixture stirred 24 hrs., treated dropwise with 3 ml. H2O with stirring, filtered, the filtrate evaporated, and the residue recrystd. from iso-Pr2O gave 2.8 g. PhCH2CH(OH)CHPhCH2OH (XXVII), m. 96-8°. XVIII treated as above gave 35% isomer of XXVII, m. 154-5° (absolute alc.). XVII (12.8 g.), 3.1 g. red P, 12 g. iodine, and 350 ml. AcOH refluxed 8 hrs., the mixture filtered, the filtrate concentrated to dryness, and the residue recrystd. from PhMe gave 8.1 g. PhCH2CH(OAc)CHPhCO2H, m. 124-6°. Cyclohexene oxide (prepared from trans-2-chlorocyclohexanol) (196.0 g.) in 500 ml. C6H6 added dropwise with stirring to II (from 272.2 g. I), the mixture stirred 48 hrs., hydrolyzed with 333 ml. concentrated HCl in 1.5 l. cold H2O, the organic layer separated, concentrated to dryness, and the residue recrystd. from PhMe gave 187.0 g. α-phenyl-α-(2-hydroxycyclohexyl)acetic acid (XXVIII), m. 153-4°. XXVIII (8.2 g.) in 300 ml. Et2O treated at 0° with excess CH2N2 in Et2O, after 2 hrs. at 0° the Et2O removed, and the residue recrystd. from petr. ether gave 7.9 g. Me ester (XXIX) of XXVIII, m. 94-5°. XXIX in 250 ml. C6H6 added dropwise with stirring to a refluxing mixture of 20 g. Celite, 15 g. P2O5, and 750 ml. C6H6, the mixture stirred and refluxed 4 hrs., cooled, filtered, the filtrate shaken with aqueous NaHCO3, dried, evaporated, and the residue distilled gave 7.8 g. dehydration product [Me α-phenyl-α-(1- cyclohexenyl)acetate or Me α-phenyl-α-(2- cyclohexenyl)acetate], b20 166-70°. XXVIII (9.3 g.), 150 ml. MeOH, and 1 ml. concentrated H2SO4 refluxed 4 hrs., the MeOH removed, the residue dissolved in Et2O, the solution extracted with aqueous NaHCO3, evaporated, and the residue recrystd. from petr. ether gave 8.4 g. lactone (XXX) of XXVIII, m. 75-7°. XXX was isolated in 90% yield in attempts to prepare the Et2NCH2CH2 ester of XXVIII by (a) stirring XXVIII Ag salt, Et2NCH2CH2Cl, and Me2CO 24 hrs., and (b) attempted transesterification of XXIX with Et2NCH2CH2OH, NaOMe, and petr. ether. XXX (8.4 g.) and 1.6 g. KOH in 150 ml. H2O refluxed 3 hrs., the cooled solution extracted with Et2O, acidified in the cold, and the precipitate recrystd. from PhMe gave 8.8 g. XXVIII, m. 153-5°. Cyclopentanecarboxaldehyde (English, et al., CA 45, 5632h) (31.0 g.) in 100 ml. C6H6 added dropwise with stirring to II (from 43.0 g. I), the mixture stirred and refluxed 4 hrs., poured into 53 ml. concentrated HCl in 500 ml.

ice H2O, the organic layer separated, the solvent removed, and the residue recrystd. from PhMe gave 53.5 g. mixture (XXXI) of α-phenyl-β-cyclopentyl-β-hydroxypropionic acids, m. 145-8°. XXXI (50 g.) heated with petr. ether (b. 60-75°) (some XXXI remained undissolved), the petr. ether containing soluble material separated, evaporated, and the residue recrystd. from petr. ether gave 35.5 g. of 1 component, m. 130-1°. The petr. ether-insol. portion recrystd. from PhMe gave 5.6 g. other component, m. 161-2°.

IT 102371-44-8 102377-30-0
(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 102371-44-8 CAPLUS

CN Chromone, 8-[(4-dipropylaminobutyl)amino]-7-methyl-, oxime (6CI) (CA INDEX NAME)



RN 102377-30-0 CAPLUS

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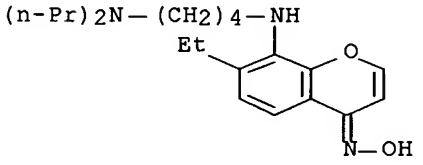

L9 ANSWER 40 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1960:128790 CAPLUS Full-text

DOCUMENT NUMBER: 54:128790

ORIGINAL REFERENCE NO.: 54:24624d-i,24625a

TITLE: Search for new antimalarials. V. Synthesis of some 8-aminoalkylamino- and 8-dialkylaminoalkylaminochromones

AUTHOR(S): Sen, A. B.; Singh, P. R.

CORPORATE SOURCE: Univ. Lucknow

SOURCE: J. Indian Chem. Soc. (1960), 37, 217-22

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

AB cf. CA 54, 9948f. In view of the antimalarial properties of some well-known amebicides, 25 title compds. (I) were synthesized as possible antimalarials. The following 7-substituted chromones (II) were prepared by the method of Joshi, et al. (CA 53, 21927a) (substituent, % yield, m.p., and m.p. of oxime given): HO, -, -, 283°; MeO, -, -, 154°; EtO, 49, 103°, 131-2°; Me, 43, 66°, 112°; Et, 46, 53-4°, 98-9°. II, on nitration at 0°, yielded the 7-substituted-8-nitrochromones (III) (data as above): HO, -, -, 291°; MeO, -, -, 267°; EtO, 93, 219°, 271-2°; Me, 96, 181°, 204°; Et, 88, 163°, 211-13°.

Reduction of III with Fe powder and H2O containing FeSO4 (Hodgson and Marsden, CA 39, 11483) gave the corresponding 7-substituted-8-aminochromones (IV) (substituent, % yield, m.p., and m.ps. of oxime and N-acetyl derivative given): HO, 41, 199°, 229-30°, 257-8°; MeO, 53, 167°, 189-91°, 233°; EtO, 38, 161-2°, 180-2°, 245°; Me, 47, 130-1°, 165-6°, 178-9°; Et, 40, 117-19°, 152-14°, 159-60°. IV refluxed with amino- and dialkylaminoalkyl bromide hydrobromides in dry C6H6 containing excess of anhydrous K2CO3 gave I, isolated as hydrochlorides (R, R', n, % yield, and m.ps. of the dihydrochloride, oxime, and dipicate, resp., given): HO, H, 1, 61, 280°, 168-9°, 202°; MeO, H, 1, 76, 297-8°, 157-8°, 178-9°; EtO, H, 1, 70, 276-7°, 153°, 181°; Me, H, 1, 69, 201°, 141°, 160-1°; Et, H, 1, 57, 189-90°, 132-3°, 152-3°; HO, Et, 1, 52, 251-2°, 156-7°, 177°; MeO, Et, 1, 61, 273-4°, 154°, 165-6°; EtO, Et, 1, 65, 243°, 147-8°, 141-2°; Me, Et, 1, 64, 221-2°, 122-3°, 128°; Et, Et, 1, 70, 167-8°, 109-11°, 137°; HO, Pr, 1, 57, 249-50°, 147-8°, 157°; MeO, Pr, 1, 64, 263°, 131-2°, 138-9°; EtO, Pr, 1, 53, 267°, 141-2°, 161-2°; Me, Pr, 1, 49, 209°, 117°, 153°; Et, Pr, 1, 51, 199-200°, 103-4°, 129-30°; HO, Et, 3, 49, 224-5°, 119-20°, 156-7°; MeO, Et, 3, 58, 209-10°, 100-1°, 137; EtO, Et, 3, 60, 177°, 109°, 122-3°; Me, Et, 3, 45, 146°, 89-90°, 126°; Et, Et, 3, 52, 131-2°, 78°, 128-9°; HO, Pr, 3, 59, 227°, 101-3°, 144°; MeO, Pr, 3, 60, 202-4°, 76°, 127-8°; EtO, Pr, 3, 53, 122-3°, 81-2°, 136-7°; Me, Pr, 3, 46, 118-19°, 63°, 128°; and Et, Pr, 3, 48, 109-11°, 46°, 131°. δ-Dipropylaminobutyl bromide hydrobromide and δ-diethylaminobutyl bromide hydrobromide were prepared in 53 and 67% yields, resp., by the method of Cortese (CA 30, 29229) and m. 93° and 117°, resp.

IT 100128-04-9 100138-36-1 100138-65-6
100705-82-6 100706-15-8 100951-92-6
101437-16-5 101437-25-6 101867-86-1
101867-93-0 101867-94-1 101867-95-2
101889-35-4 101889-36-5 101889-40-1
101889-41-2 102371-44-8 102371-47-1
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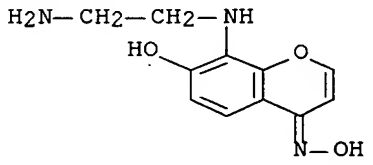
(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 100128-04-9 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)



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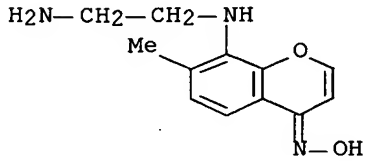

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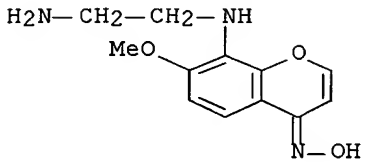

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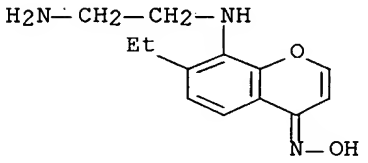

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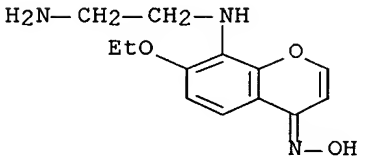

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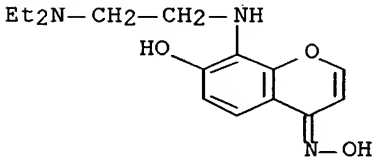

RN 101437-16-5 CAPLUS
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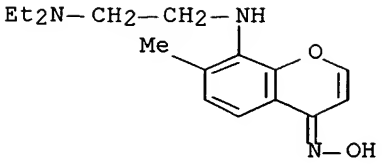

RN 101437-25-6 CAPLUS
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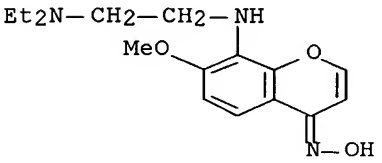

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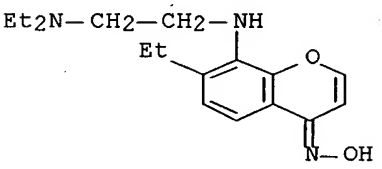

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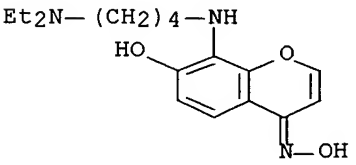

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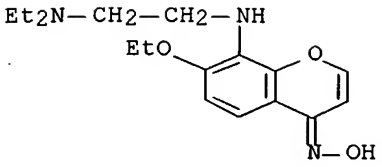

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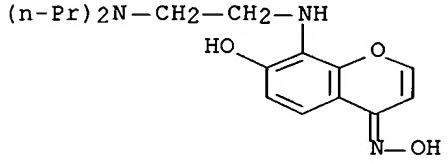

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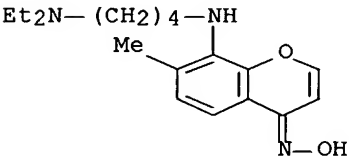

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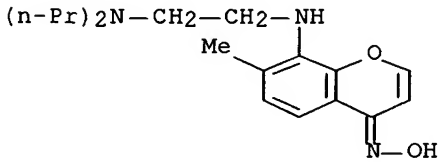

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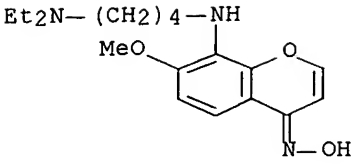
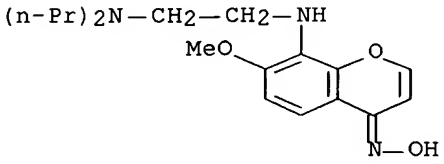
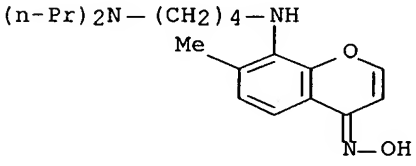


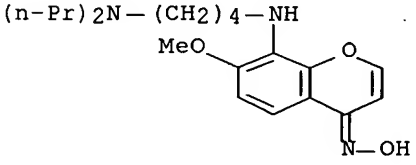
RN 101889-41-2 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



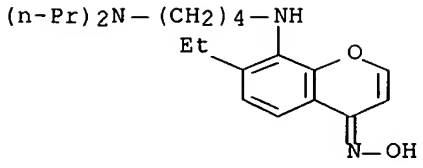
RN 102371-44-8 CAPLUS
CN Chromone, 8-[(4-dipropylaminobutyl)amino]-7-methyl-, oxime (6CI) (CA INDEX NAME)



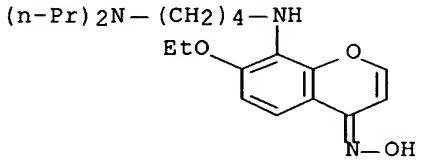
RN 102371-47-1 CAPLUS
CN Chromone, 8-[(4-di-propylaminobutyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



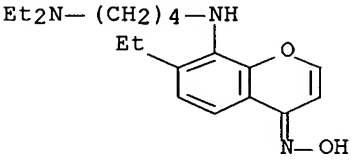
RN 102377-30-0 CAPLUS
CN Chromone, 8-[(4-dipropylaminobutyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



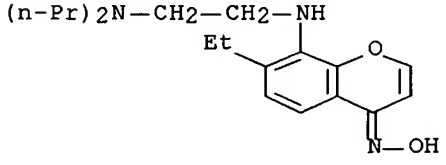
RN 102377-32-2 CAPLUS
CN Chromone, 8-[(4-dipropylaminobutyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



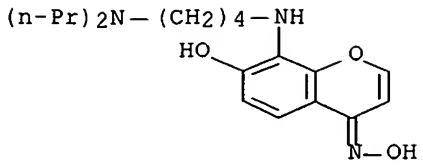
RN 102659-19-8 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



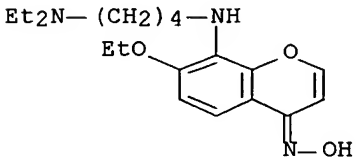
RN 102659-20-1 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



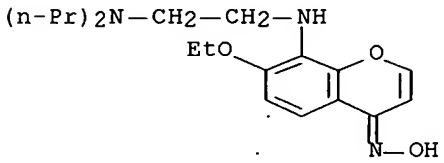
RN 102659-21-2 CAPLUS
CN Chromone, 8-[(4-dipropylaminobutyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)



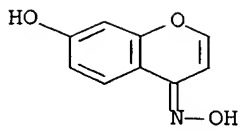
RN 112441-18-6 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



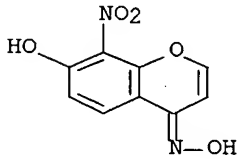
RN 112441-19-7 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



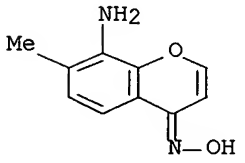
IT 98589-51-6P, Chromone, 7-hydroxy-, oxime 99066-83-8P, Chromone, 7-hydroxy-8-nitro-, oxime 99071-78-0P, Chromone, 8-amino-7-methyl-, oxime 99072-21-6P, Chromone, 8-amino-7-methoxy-, oxime 99184-95-9P, Chromone, 7-methoxy-, oxime 99361-54-3P, Chromone, 7-methyl-8-nitro-, oxime 99361-65-6P, Chromone, 7-methoxy-8-nitro-, oxime 99842-75-8P, Chromone, 7-ethyl-, oxime 99843-11-5P, Chromone, 7-ethoxy-, oxime 99985-18-9P, Chromone, 7-ethyl-8-nitro-, oxime 99985-45-2P, Chromone, 7-ethoxy-8-nitro-, oxime 100060-31-9P, Chromone, 8-amino-7-ethyl-, oxime 100060-71-7P, Chromone, 8-amino-7-ethoxy-, oxime 103261-60-5P, Chromone, 7-methyl-, oxime 108128-33-2P, Chromone, 8-amino-7-hydroxy-, oxime
RL: PREP (Preparation)
(preparation of)
RN 98589-51-6 CAPLUS
CN Chromone, 7-hydroxy-, oxime (6CI) (CA INDEX NAME)



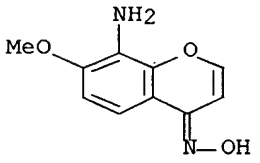
RN 99066-83-8 CAPLUS
CN Chromone, 7-hydroxy-8-nitro-, oxime (6CI) (CA INDEX NAME)



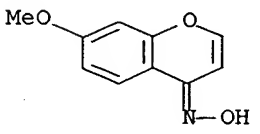
RN 99071-78-0 CAPLUS
CN Chromone, 8-amino-7-methyl-, oxime (6CI) (CA INDEX NAME)



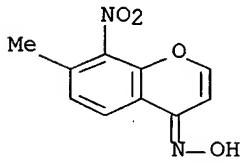
RN 99072-21-6 CAPLUS
CN Chromone, 8-amino-7-methoxy-, oxime (6CI) (CA INDEX NAME)



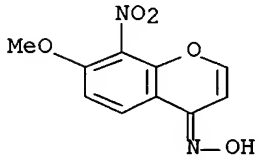
RN 99184-95-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-, oxime (9CI) (CA INDEX NAME)



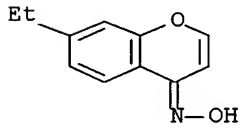
RN 99361-54-3 CAPLUS
CN Chromone, 7-methyl-8-nitro-, oxime (6CI) (CA INDEX NAME)



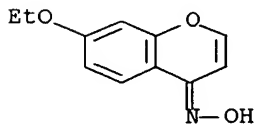
RN 99361-65-6 CAPLUS
CN Chromone, 7-methoxy-8-nitro-, oxime (6CI) (CA INDEX NAME)



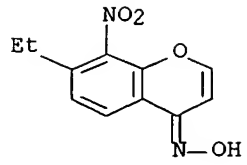
RN 99842-75-8 CAPLUS
CN Chromone, 7-ethyl-, oxime (6CI) (CA INDEX NAME)



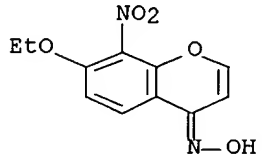
RN 99843-11-5 CAPLUS
CN Chromone, 7-ethoxy-, oxime (6CI) (CA INDEX NAME)



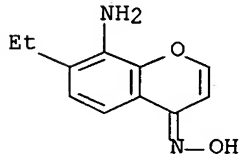
RN 99985-18-9 CAPLUS
CN Chromone, 7-ethyl-8-nitro-, oxime (6CI) (CA INDEX NAME)



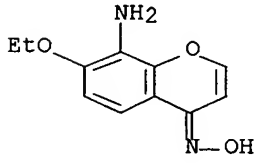
RN 99985-45-2 CAPLUS
CN Chromone, 7-ethoxy-8-nitro-, oxime (6CI) (CA INDEX NAME)



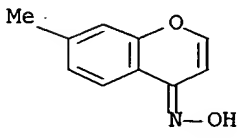
RN 100060-31-9 CAPLUS
CN Chromone, 8-amino-7-ethyl-, oxime (6CI) (CA INDEX NAME)



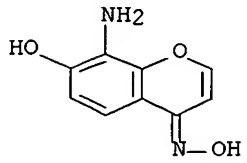
RN 100060-71-7 CAPLUS
CN Chromone, 8-amino-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



RN 103261-60-5 CAPLUS
CN Chromone, 7-methyl-, oxime (6CI) (CA INDEX NAME)



RN 108128-33-2 CAPLUS
CN Chromone, 8-amino-7-hydroxy-, oxime (6CI) (CA INDEX NAME)

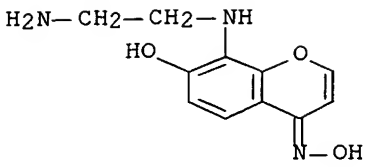


L9 ANSWER 41 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1960:128789 CAPLUS Full-text
DOCUMENT NUMBER: 54:128789
ORIGINAL REFERENCE NO.: 54:24624b-d
TITLE: Reactions of chlorinated furanidines. II. Synthesis of substituted 2-alkoxy-3-chlorotetrahydrofurans
AUTHOR(S): Kratochvil, M.
CORPORATE SOURCE: Vojenska tech. akad. A. Zapotockeho, Brno, Czech.
SOURCE: Collection of Czechoslovak Chemical Communications (1960), 25, 1351-8
CODEN: CCCCAK; ISSN: 0010-0765
DOCUMENT TYPE: Journal
LANGUAGE: Russian

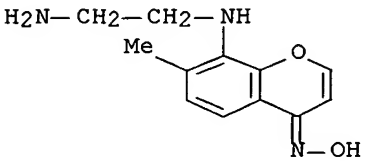
AB cf. CA 52, 16329f. Allowing to react at -5 to 0° 0.5 mole 2,3-dichlorotetrahydrofuran (I) with 0.05 mole ethylene oxide in 50 ml. dry CCl4 in the presence of 0.02-0.03 g. anhydrous ZnCl2 gives 86-8% title compds. (II) (alkyl, b.p./mm., nD20, and d20 given): CH2CH2Cl, 110.5°/15, 1.4757, 1.2816; CHMeCH2Cl, 76-6.5°/2, 1.4676, 1.2190; CH(CH2Cl)2, 121-2°/6, 1.4902, 1.3533; CH2CHClCH2Cl, 118-20°/6, 1.4906, 1.3586; CH(CH2OH)CH2Cl, 120-2°/3, 1.4882, 1.3315, CH2CHClCH2Cl, 163.5-4.0°/2, 1.4939, 1.3410; CH2CH(OH)CH2Cl, 122-4°/3, 1.4890, 1.3285; (CH2)3Cl, 110-12°/12, 1.4711, 1.2308. II (R = CH2CH:CH2) was obtained in 104-g. yield by adding at 45-50° 0.05 g. ZnCl2 and 58 g. allyl alc. to 141 g. I, refluxing the mixture 3 hrs. to 70-5°, and working up as usual to give a liquid, b14 78-9°, nD20 1.4609, d20 1.1256. Infrared spectra were charted.

IT 100128-04-9 100138-36-1 100138-65-6
100705-82-6 100706-15-8 100951-92-6
101437-16-5 101437-25-6 101867-86-1
101867-93-0 101867-94-1 101867-95-2
101889-35-4 101889-36-5 101889-40-1
101889-41-2 102371-47-1 102377-32-2
102659-19-8 102659-20-1 102659-21-2
112441-18-6 112441-19-7
(Derived from data in the 6th Collective Formula Index (1957-1961))

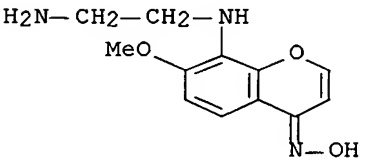
RN 100128-04-9 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)



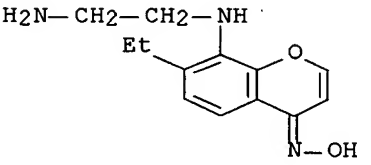
RN 100138-36-1 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-methyl-, oxime (6CI) (CA INDEX NAME)



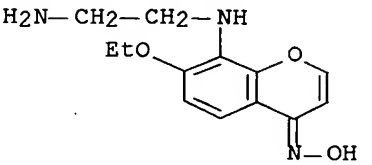
RN 100138-65-6 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



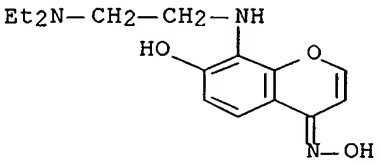
RN 100705-82-6 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



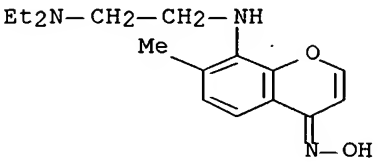
RN 100706-15-8 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



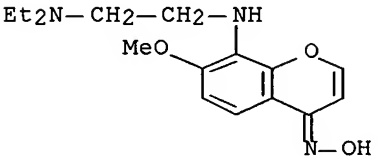
RN 100951-92-6 CAPLUS
CN Chromone, 8-[(2-diethylaminoethyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)



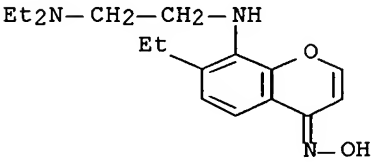
RN 101437-16-5 CAPLUS
CN Chromone, 8-[(2-diethylaminoethyl)amino]-7-methyl-, oxime (6CI) (CA INDEX NAME)



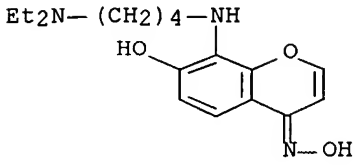
RN 101437-25-6 CAPLUS
CN Chromone, 8-[(2-diethylaminoethyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



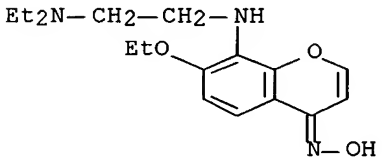
RN 101867-86-1 CAPLUS
CN Chromone, 8-[(2-diethylaminoethyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



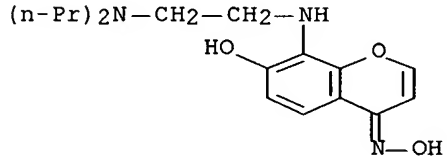
RN 101867-93-0 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)



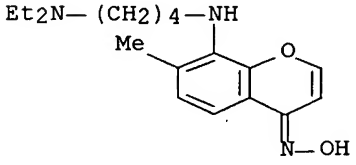
RN 101867-94-1 CAPLUS
CN Chromone, 8-[(2-diethylaminoethyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



RN 101867-95-2 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)

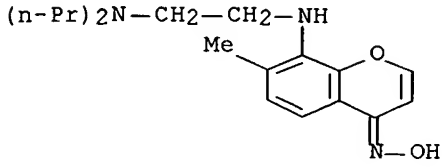


RN 101889-35-4 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-methyl-, oxime (6CI) (CA INDEX NAME)

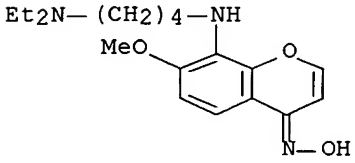


RN 101889-36-5 CAPLUS

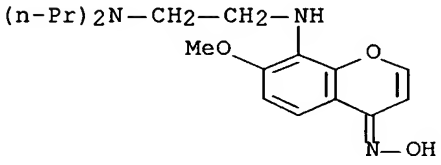
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-methyl-, oxime (6CI) (CA INDEX NAME)



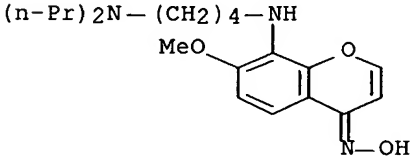
RN 101889-40-1 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



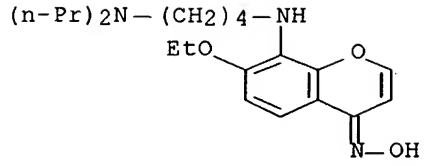
RN 101889-41-2 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



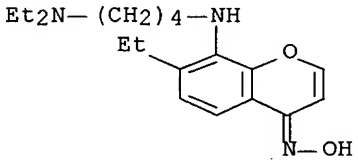
RN 102371-47-1 CAPLUS
CN Chromone, 8-[(4-di-propylaminobutyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



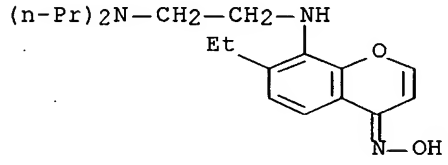
RN 102377-32-2 CAPLUS
CN Chromone, 8-[(4-dipropylaminobutyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



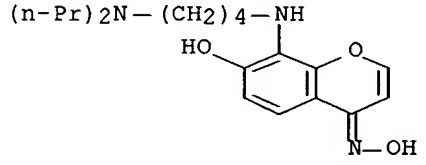
RN 102659-19-8 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



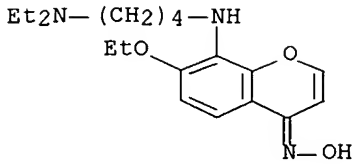
RN 102659-20-1 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



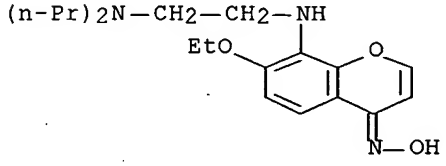
RN 102659-21-2 CAPLUS
CN Chromone, 8-[(4-dipropylaminobutyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)



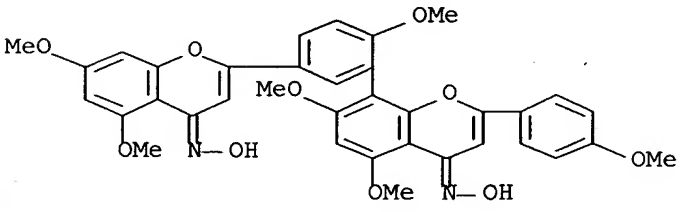
RN 112441-18-6 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



RN 112441-19-7 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 42 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1960:86472 CAPLUS Full-text
DOCUMENT NUMBER: 54:86472
ORIGINAL REFERENCE NO.: 54:16449b-d
TITLE: Synthesis of ginkgetin tetramethyl ether
AUTHOR(S): Nakazawa, Koichi
CORPORATE SOURCE: Coll. Pharmacy, Gifu
SOURCE: Chem. & Pharm. Bull. (Tokyo) (1959), 7, 748-9
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB The synthesis was given for methylated biflavonyl (I), m. 238° (HCONMe2), which was identical with the title compound (CA 44, 9441i). 2-Acetyl-3,5-dimethoxyphenyl 3-iodoanisate and 2-acetyl-6-iodo-3,5- dimethoxyphenyl anisate were isomerized to the ketones in pyridine by KOH (yields of 40% and 78%, resp.). The ketones were cyclized to the flavones (91% and 85% yields, resp.) in H2SO4 and HOAc and the iodinated flavone (1 mole each) was converted to 28% I by heating 8 hrs. with an equal weight of Cu powder in boiling HCONMe2. I was slightly soluble in MeOH, EtOH, and dioxane, and gave a dioxime (m. 252°).
IT 107225-53-6P, Ginkgetin, tetra-O-methyl-, dioxime
RL: PREP (Preparation)
(preparation of)
RN 107225-53-6 CAPLUS
CN 3''',8-Biflavone, 4',4''',5,5'',7,7''-hexamethoxy-, dioxime (7CI) (CA INDEX NAME)

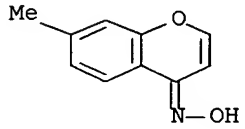


L9 ANSWER 43 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1959:122072 CAPLUS Full-text
DOCUMENT NUMBER: 53:122072
ORIGINAL REFERENCE NO.: 53:21909a-g
TITLE: 4-Chromanones. III. Alkylation and bromination of chromanones. Transformation into chromones
AUTHOR(S): Colonge, J.; Guyot, A.
CORPORATE SOURCE: Fac. sci., Lyon
SOURCE: Bulletin de la Societe Chimique de France (1958) 329-34
CODEN: BSCFAS; ISSN: 0037-8968
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

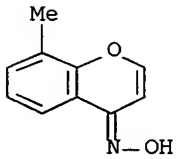
AB By reaction with tert-C5H11ONa [in C6H4(Me)2] and MeI (cf. Vavon and Conia, C.A. 41, 721a), and purification via the semicarbazones, the following compds. were obtained from, resp., I, IV, and X: [m.p., b.p. (pressure in mm.); semicarbazone m.p. given]: VII (-, 154°(40); 238°]; VIII [34, 156°(25); 233°], and IX [42.5, 185-95°(7); 271°]. By refluxing the chromanones in Et2O with 1.1 moles Br, the 3-bromo-derivs. of the following chromanones were obtained (chromanone, % yield, m.p. given): I, 78, 70°; VII, 57, 40°; IV, 77, 74°; III, 48, 89°; II, 64, 90°; VI, 60, 93°; V, 58, 153°; XI, 77, 116°; and X, 76, 113°. With 2.2 moles Br, 3,3-dibromo derivs. were obtained (as above): I, 98, 72°; IV, 94 (yield on monobromocompd.), 119°; and II, 66 (yield on monobromocompd.), 106°. The bromocompds. attacked skin and mucosas. To determine Br, the substances had to be refluxed for 7 hrs. with 0.5N KOH in glycol and the Br ion titrated potentiometrically (Lange and Berger, C.A. 25, 1179). By refluxing 48 g. 3-bromo-4-chromanone (XII) and 100 g. PhNMe2, 55% chromone (XIII) (m. 57°, oxime m. 184°) was obtained. Stirring at room temperature for 25 hrs. 20 g. XII, 13 g. HNEt2, and 50 ml. H2O yielded 72% 3-diethylamino-4-chromanone (m. 76°) which, refluxed with HCl, gave XIII, which was also obtained in 65% yield by (CO2H)2 hydrolysis of 3-(piperidino)-4-chromanone (m. 117°, obtained in 86% yield from XII and piperidine in petr. ether). From 3-bromo-6-methyl-4-chromanone, 6-methyl-3-(piperidino)-4-chromanone (m. 131°) was obtained in 65% yield. With (CO2H)2 it gave a 57% yield of 6-methylchromone (m. 88°, oxime m. 174°). Action of HNEt2 on 3-bromo-7-methyl-4-chromanone gave directly 58% 7-methylchromone (m. 73°, oxime m. 185°). From 3-bromo-8-methyl-4-chromanone, HNEt2 and (CO2H)2 hydrolysis yielded 56% 8-methylchromone (m. 84°, oxime m. 107°). Reaction of Zn powder with alc. and 2,3-dibromo-4-chromanone (XIV) [Arndt, Ber. 58, 1612(1925)] yielded XIII, which was not obtained from 3,3-dibromo-4-chromanone (XV). 3-Bromochromone (XVI) (m. 93°) could be obtained: (a) from XIV by addition of piperidine in Et2O; (b) from XV by HNEt2, HNMe2 or, best, piperidine addition Hydrogenation (Pd) of XVI yielded XII. The product, m. 65°, obtained by Arndt (loc. cit.) was not XVI (infrared spectrum). XVI with piperidine yielded 3-(piperidino)chromone (m. 124°) which, with (CO2H)2, gave XIII. Addition of 5.5 g. Br to 5 g. 6-methylchromone yielded 27% 2,3-dibromo-6-methyl-4-chromanone (m. 94°), which gave with piperidine: (a) in Et2O solution, 60% 3-bromo-6-methylchromone (m. 101°); (b) directly (with cooling), 52% 6-methyl-3-(piperidino)chromone (m. 128.5°). Similarly were obtained 2,3-dibromo-7-methyl-4-chromanone (yield 34%, m. 89°), and 3-bromo-7-methylchromone (yield 64%, m. 107°). Addition of Br to 8-methylchromone in Et2O gave a product, m. 112°, giving with HNEt2 3-bromo-8-methylchromone (m. 114°), also obtained from 3,3-dibromo-8-methyl-4-chromanone and HNEt2 (or piperidine).

IT 103261-60-5P, Chromone, 7-methyl-, oximes 103261-61-6P, Chromone, 8-methyl-, oximes 103264-01-3P, Chromone, 6-methyl-, oximes
RL: PREP (Preparation)

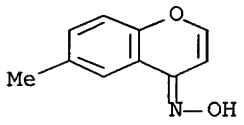
(preparation of)
RN 103261-60-5 CAPLUS
CN Chromone, 7-methyl-, oxime (6CI) (CA INDEX NAME)



RN 103261-61-6 CAPLUS
CN Chromone, 8-methyl-, oxime (6CI) (CA INDEX NAME)



RN 103264-01-3 CAPLUS
CN Chromone, 6-methyl-, oxime (6CI) (CA INDEX NAME)

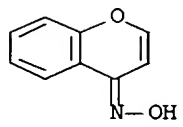


L9 ANSWER 44 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1959:122071 CAPLUS Full-text
DOCUMENT NUMBER: 53:122071
ORIGINAL REFERENCE NO.: 53:21908d-i,21909a
TITLE: 4-Chromanones. II. Cyclodehydration of 3-aryloxyalkanoic acids, tertiary chromanols and chromenes
AUTHOR(S): Colonge, J.; Guyot, A.
CORPORATE SOURCE: Fac. sci., Lyon
SOURCE: Bulletin de la Societe Chimique de France (1958) 325-8
CODEN: BSCFAS; ISSN: 0037-8968
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 53:122071
AB cf. C.A. 52, 6280h. 3-Phenoxypropionic acid (85 g.) was dissolved in 250 ml. MePh. Water (15 ml.) was added and, slowly, 50 g. P2O5. After 1 hr. refluxing with stirring, another 50 g. P2O5 was added. After 1 hr. the MePh layer was washed with Na2CO3 solution and the aqueous one diluted with H2O and extracted with Et2O. Both solns. were washed with H2O, dried (Na2SO4) and distilled to yield 82% 4-chromanone (I), m. 38.5° (oxime, m. 138°). From 3-o-

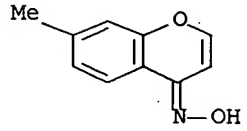
tolyloxypropionic, 3-m-tolyloxypropionic, 3-p-tolyloxypropionic, 3-o-methoxyphenyloxypropionic, 3-m-methoxyphenyloxypropionic, 3-p-methoxyphenyloxypropionic, m-phenylenebis(3-oxypropionic), p-phenylenebis(3-oxypropionic), 2-methyl-3-phenoxypropionic, 2-methyl-3-p-tolyloxypropionic, 2-methyl-3-m-tolyloxypropionic, 2-methyl-3-o-tolyloxypropionic, and 2-methyl-3-β-naphthoxypropionic acids were obtained, resp.: [% yield, m.p., b.p. (pressure in mm.); oxime m.p. given]: 8-methyl-4-chromanone (II) [81, 29.5°, 170°(42); 123°], 7-methyl-4-chromanone (III) [69, viscous oil, 151°(27), d21 1.1576, n21D 1.544; 98°], 6-methyl-4- chromanone (IV) [68, 34°, 160-2°(28); 84°], 8-methoxy-4-chromanone [25, 89°, -; 146°], 7-methoxy-4-chromanone (V) [58, 55°, -; 134°]; 6-methoxy-4-chromanone (VI) [60.5, 48°, 178-80°(23); 120°], 7-(2-carboxyethoxy)-4-chromanone [54 (in dioxane), 169°, -; 223°], 1,5-dioxa-2,3,4,6,7,8-hexahydro-4,8- anthracenedione [10(in anisole), 234° (decompose), -; dioxime decompose 300°], 3-methyl-4-chromanone (VII) [60, liquid, 154°(40), d26.5 1.131, n26.5D 1.5563; 156°], 3,6-dimethyl-4-chromanone (VIII) [66, 33°, 158-60° (30); 129°], 3,7-dimethyl-4- chromanone [53, 54°, 159-62°(30); 120°], 3,8-dimethyl-4-chromanone [58, 65°, 170-2°(40); 118°], and 3-methyl-5,6-benzo-4-chromanone (IX) [53, 42.5°, -; 110°]. By the method of Bachman and Levine (C.A. 42, 169h) 3-β-naphthoxypropionitrile and 3-α-naphthoxypropionitrile were converted into, resp., 5,6-benzo-4-chromanone (X) [82, 44°, 160-1°(2.5); 112°], and 7,8-benzo-4-chromanone (XI) [68 105°, -; 137°]. By Grignard reaction with MeMgI and hydrolysis, the chromanones were transformed into the resp. 4-methyl-4-chromanols (chromanone, % yield, m.p.): I, 70, 107°; II, 97, 72°; III, 94, 80°; IV, 94, 116°; V, 79, 61°; VI, 79, 71°; X, 95, 124°; and XI, 77, 87°. By refluxing (and separating dist. H2O) in C6H6 solution with a few dg. anhydrous CuSO4, these tertiary chromanols yielded 4-methyl-3-chromenes, separated by filtration and C6H6 evaporation Thus the following 4-methyl-3-chromenes were obtained [% yield, b.p. (pressure in mm.), dt (t in °C), ntD given]: unsubstituted [83, 102°(8), 1.074(23°), 1.590]; 6-methyl- [86, 134°(22), 1.041(24°), 1.573]; 7-methyl- [86, 123°(16), 1.054(21°), 1.574]; 8-methyl- [79, 128°(22), 1.054(20°), 1.572]; 5,6-benzo- [58, 150°(3), 1.195(17°), 1.659]; 7,8 benzo- [58, 170°(7), 1.194(19°), 1.657]; 6-methoxy- [62, 147°(6), 1.140(19°), 1.576]; and 7-methoxy- [60, 144°(6), 1.143(19°), 1.575]. 4-Ethyl-3-chromene (b10 125°, d23 1.076, nD 1.569) was prepared directly from I and EtMgI in 70% yield, as the alc. decomposed

IT 61348-46-7 103261-60-5 103261-61-6
103264-01-3
(Derived from data in the 6th Collective Formula Index (1957-1961))

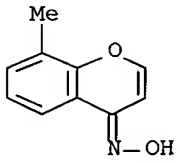
RN 61348-46-7 CAPLUS
CN 4H-1-Benzopyran-4-one, oxime (9CI) (CA INDEX NAME)



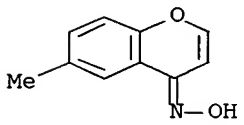
RN 103261-60-5 CAPLUS
CN Chromone, 7-methyl-, oxime (6CI) (CA INDEX NAME)



RN 103261-61-6 CAPLUS
CN Chromone, 8-methyl-, oxime (6CI) (CA INDEX NAME)

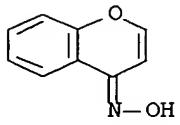


RN 103264-01-3 CAPLUS
CN Chromone, 6-methyl-, oxime (6CI) (CA INDEX NAME)

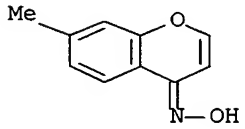


L9 ANSWER 45 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1959:122070 CAPLUS Full-text
DOCUMENT NUMBER: 53:122070
ORIGINAL REFERENCE NO.: 53:21907i,21908a-d
TITLE: Biflavonyls, a new class of natural product. The structures of ginkgetin, isoginkgetin, and sciadopitysin
AUTHOR(S): Baker, W.; Finch, A. C. M.; Ollis, W. D.; Robinson, K. W.
CORPORATE SOURCE: Univ. Bristol, UK
SOURCE: Proc. Chem. Soc. (1959) 91-2
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.
AB cf. Nakazawa, Yakugaku Zasshi 61, 228(1941). Ginkgetin (I) (R = H, R' = Me) (Ia) m. 342-4°, and isoginkgetin (I, R = Me, R' = H) (II), m. 355°, were isolated from Ginkgo biloba leaves. Methylation of Ia and II gave the same tetra-Me ether (III) while demethylation gave the same hexahydric phenol. Ia and II gave different tetra-acetates. Trimethylation of sciadopitysin (I, R = R' = Me) (IV) (Kariyone and Kawano, C.A. 50, 16759g) gave III. III with alkaline H2O2 gave p-anisic acid (V), 2,4,6-HO(MeO)2C6H2CO2H and C12H4(OMe)3(OH)(CO2H)2. Similarly, IV gave 4-methoxyisophthalic acid (VI) and V. Alkaline hydrolysis of IV gave C23H14O5(OMe)2 and 2,6,4-(HO)2(MeO)C6H2Ac. Infrared and ultraviolet spectra of IV placed hydroxyls in the 5''- and 7''-positions. II with alkaline H2O2 gave V and VI. Similarly, I gave VI and p-HOC6H4CO2H. Infrared and ultraviolet spectra of I placed hydroxyls at positions 5, 5'', and 7''. The 3',8''-biflavonoid structure was preferred on the bases of mechanism of biosynthesis, ease of methylation, and lack of optical activity.
IT 61348-46-7 103261-60-5 103261-61-6

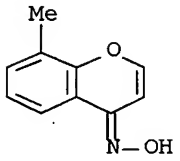
103264-01-3
(Derived from data in the 6th Collective Formula Index (1957-1961))
RN 61348-46-7 CAPLUS
CN 4H-1-Benzopyran-4-one, oxime (9CI) (CA INDEX NAME)



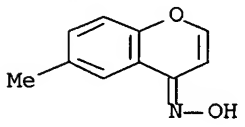
RN 103261-60-5 CAPLUS
CN Chromone, 7-methyl-, oxime (6CI) (CA INDEX NAME)



RN 103261-61-6 CAPLUS
CN Chromone, 8-methyl-, oxime (6CI) (CA INDEX NAME)



RN 103264-01-3 CAPLUS
CN Chromone, 6-methyl-, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 46 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1959:17230 CAPLUS Full-text
DOCUMENT NUMBER: 53:17230
ORIGINAL REFERENCE NO.: 53:3203e-g
TITLE: Flavonoids of the leaves of Coniferae and allied plants. II. Flavonoids from the leaves of Cycas revoluta and Cryptomeria japonica var. araucarioides

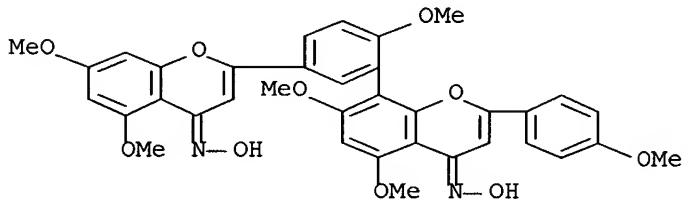
AUTHOR(S): Kariyone, Tatsuo; Sawada, Tokunosuke
SOURCE: Yakugaku Zasshi (1958), 78, 1013-15
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB Extraction of 5 kg. dried leaves of C. revoluta in a similar way yielded 4.1 g. sotetsuflavone (I), C31H20O10.H2O, columns, m. 264-5° (decomposition) (MeOH). I gives penta-Ac derivative, needles, m. 233-4°; tri-Me derivative (II), m. 281-2° (identical with kayaflavone mono-Me ether by mixed m.p.); II diacetate, m. 228-30° (identical with mono-Me kayaflavone diacetate by mixed m.p.); penta-Me derivative (III), m. 220-1° (identical with trimethylkayaflavone and tetramethylginkgetin by mixed m.p.); III dioxime, m. 249-50° (identical with trimethylkayaflavone dioxime by mixed m.p.); penta-Et derivative, m. 234-5° (identical with tetraethylginkgetin and triethylkayaflavone by mixed m.p.). Demethylation of I and its acetylation yielded demethylkayaflavone hexaacetate, m. 239-40°. Thus, I is monodemethylginkgetin. Extraction of dried leaves of C. japonica var. araucarioides in a similar way gives kayaflavone, m. 314-5° (decomposition), sciadopitysin, m. 286-7° (decomposition), and I, m. 263-4° (decomposition).

IT 107225-53-6, Sotetsuflavone, penta-O-methyl-, dioxime (structure of)

RN 107225-53-6 CAPLUS

CN 3''',8-Biflavone, 4',4''',5,5'',7,7''-hexamethoxy-, dioxime (7CI) (CA INDEX NAME)



L9 ANSWER 47 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1959:17229 CAPLUS Full-text
DOCUMENT NUMBER: 53:17229
ORIGINAL REFERENCE NO.: 53:3203a-e
TITLE: Flavonoids of the leaves of Coniferae and allied plants. I. Flavonoid from the leaves of Torreya nucifera
AUTHOR(S): Kariyone, Tatsuo; Sawada, Tokunosuke
SOURCE: Yakugaku Zasshi (1958), 78, 1010-13
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

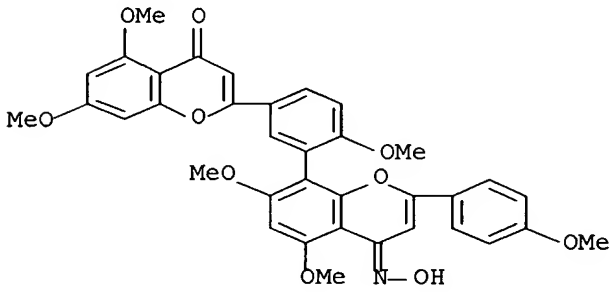
AB Dried leaves (10 kg.) extracted with hot MeOH, the MeOH extract filtered while hot, cooled, the waxy precipitate filtered off, washed with 2% NaOH, the NaOH washing acidified with dilute H2SO4, the precipitate filtered off, washed with C2HCl3, taken up in C5H5N, and H2O added yielded 0.15% kayaflavone (I), C33H24O10.H2O, needles, m. 314-15° (decomposition)(MeOH). I (0.3 g.) and 1.5 g. Ac2O treated with 1-2 drops concentrated H2SO4, the solution poured into H2O, and the precipitate recrystd. from EtOH gave tri-Ac derivative (II) of I, needles, m. 190-1°. I (0.2 g.) in 50 mL. Me2CO, 2 g. MeI, and 2 g. K2CO3 refluxed 1 h., the product concentrated, and recrystd. from Me2CO gave a mono-

Me derivative (III) of I, m. 281-2°, identical with ginkgetin di-Me ether by mixed m.p. Acetylation of III with Ac2O and concentrated H2SO4 gave a diacetate of III, needles, m. 230°, identical with dimethylginkgetin diacetate by mixed m.p. Methylation of I with Me2SO4 gave tri-Me derivative (IV) of I, m. 220-1°, identical with ginkgetin tetra-Me ether by mixed m.p. IV (0.1 g.), 0.1 g. NH2OH.HCl, 50 mg. AcONa, and 3 mL. C5H5N refluxed 5 h., cooled, dilute AcOH added, and the precipitate recrystd. from EtOH gave IV oxime, prisms, m. 250°. Similarly is prepared triethylkayaflavone, columns, m. 236-7°. I (0.5 g.) in a small amount of PhOH and 20 mL. HI heated 2 h. at 130-40°, the solution diluted with H2O, and the precipitate recrystd. from MeOH gave demethylkayaflavone (V), prisms, m. above 330°. Acetylation of V gave V acetate, needles, m. 239-40°. Alkali fusion of I yielded AcOH, p-HOC6H4CO2H, and phloroglucinol. Thus, I is ginkgetin mono-Me ether.

IT 124270-14-0P, Kayaflavone, tri-O-methyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 124270-14-0 CAPLUS

CN Kayaflavone, tri-O-methyl-, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 48 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:	1958:25516 CAPLUS Full-text
DOCUMENT NUMBER:	52:25516
ORIGINAL REFERENCE NO.:	52:4622f-i,4623a-i,4624a-e
TITLE:	Chemistry of fungi. XXVII. Structure of fulvic acid from Carpenteles brefeldianum
AUTHOR(S):	Dean, F. M.; Eade, R. A.; Moubasher, R.; Robertson, Alexander
CORPORATE SOURCE:	Univ. Liverpool, UK
SOURCE:	Journal of the Chemical Society (1957) 3497-510 CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE:	Journal
LANGUAGE:	Unavailable

GI For diagram(s), see printed CA Issue.

AB cf. C.A. 51, 3507e. Evidence is presented that fulvic acid (I), the yellow acidic metabolite of C. brefeldianum, has the structure indicated. C. brefeldianum was inoculated on Raulin-Thom medium by the method of Oxford, et al. (C.A. 29, 58378), except that a temperature of 26° was used to isolate satisfactory yields of I from the culture medium after an appreciably shorter period (28 days) of growth. The culture fluid acidified, extracted with EtOAc, and the product purified by repeated crystallization from anhyd dioxane (II) yielded I, pale yellow prisms, m. 244° (vigorous decomposition) (softening above 200°), giving a green ferric reaction in alc., λ 225, 318, 343 mμ (log ε 4.47, 4.07, 4.05), with characteristic infrared spectrum. I was recovered from its solution in H2SO4.H2O after being kept overnight and then

poured on ice. Anhydrofulvic acid (III) (0.2 g.) refluxed 1 hr. with 50 ml. 2N H_2SO_4 and the solution diluted gave 0.13 g. I, m. 244° (II), identified spectroscopically. I (1.5 g.) and 2N aqueous NaOH distilled slowly during 0.5 hr. with the addition of H_2O to maintain the original volume, and the distillate worked up gave 2,4-$(O_2N)_2C_6H_3NHN{:}CMe_2$, yellow needles, m. 124-5°. Acidification of the residual alkaline liquor gave a precipitate which was removed and distillation of the filtrate yielded 0.94 equivalent volatile acids, including HOAc, identified as 2-methylbenzimidazole (IV), m. 173-4°. I (0.25 g.) refluxed 0.5 hr. with 30 ml. 10% aqueous NaOH, the cooled mixture acidified carefully with 2N aqueous H_2SO_4, and warmed gently while a slow stream of N was passed into the mixture and then into $Ba(OH)_2$ solution showed by titration that 0.812 mole of CO_2 had been formed. In similar expts. with III, 0.850 mole CO_2 was formed. Methylation of I with CH_2N_2 or with Me_2SO_4 and Na_2CO_3 (loc. cit.) gave Me di-O-methylfulvate, prisms, m. 186-7° (decomposition) (aqueous II), λ 230, 282, 300 mμ (log ε 4.41, 4.08, 3.99), inflection at 248 mμ (log ε 4.04), insol. in dilute NaOH, and giving a neg. ferric reaction. The solution obtained by heating 1.0 g. Me anhydrodi-O-methylfulvate (VI) 0.75 hr. with 20 ml. MeOH and 20 ml. 2N H_2SO_4, diluted with 40 ml. H_2O, concentrated in vacuo to 35 ml., the precipitate (0.1 g.) filtered off, and the filtrate stored gave 0.85 g. V, m. 191° (decomposition). MeOH (5 ml.) containing NaOMe (from 0.2 g. Na) added to 1.0 g. VI in 10 ml. warm absolute MeOH gave Me tri-O-methylfulvate (VII), cubes, m. 176-7° (C_6H_6-petr. ether), giving no ferric reaction. VI (1.0 g.) and 50 ml. 3% HCl-absolute MeOH gave 0.8 g. VII, m. 175-6° (decomposition), λ 230, 283, 300 mμ (log ε 4.42, 4.09, 4.01). Substitution of EtOH for MeOH in either of the above 2 prepns. gave Me O-ethyldi-O-methylfulvate (VIII), plates, m. 208-9° (decomposition) (EtOH). I (8.0 g.) in 1 1.70% HOAc boiled 10 min. and cooled gave 6.4 g. III, yellow, m. 245-6° (darkening near 235°) (anhydrous or aqueous II), giving a deep green ferric reaction, λ 233, 341, 387 mμ (log ε 4.28, 4.03, 4.34), and characterized by its infrared spectrum. Finely powdered III (10 g.) in 200 ml. Et_2O containing 20 ml. MeOH treated with Et_2O-CH_2N_2 [from 30 g. $Me(NO)NCONH_2$] gave 9.8 g. VI, faint yellow prisms, m. 193-4°, mixed m.p. with V depressed to 170°, mol. weight (micro Rast) 324, λ 235, 257, 318, 348 mμ (log ε 4.12, 4.09, 4.13, 4.20), having a characteristic infrared spectrum. Me_2SO_4 (60 ml.) in 30 ml. MeOH added to a stirred solution of 10 g. III in 200 ml. N aqueous Na_2CO_3, the mixture treated gradually with 400 ml. 2N Na_2CO_3 (the temperature kept below 40°), and the solid collected after 2 hrs., triturated with dilute aqueous NaOH, and crystallized from MeOH gave 9.3 g. VI, m. 193°. Acidification of the alkaline liquors gave a precipitate partly soluble in aqueous $NaHCO_3$, from which solution was obtained 0.1 g. anhydrodi-O-methylfulvic acid, pale yellow plates, m. 219-20° (decomposition) (aqueous MeOH). When sublimed at 180°/0.005 mm., V, VII, and VIII afforded VI, m. 193°. VI (0.5 g.) and 0.5 g. piperonal in 20 ml. warm MeOH containing NaOMe (from 0.2 g. Na) gave, after 2 weeks, 0.4 g. piperonylidene derivative, orange needles, m. 217-18° (decomposition) (MeOH). III (1.0 g.) and $MeCHN_2$ gave 0.5 g. Et anhydrodi-O-ethylfulvate, yellow plates, m. 149-51° (decomposition). VI (3 g.) in 250 ml. EtOAc shaken with 3 g. 10% Pd-C absorbed 240-70 ml. H; the mixture filtered, and the filtrate concentrated to 50 ml. gave 2.3 g. Me deoxydi-O-methylfulvate, prisms, m. 199-200° (EtOAc), giving a neg. ferric reaction, λ 230, 282, 303 mμ (log ε 4.40, 4.08, 4.00). VI (0.5 g.) and 0.5 g. o-$C_6H_4(NH_2)_2$ in 8 ml. EtOH containing 1 ml. HOAc refluxed 1 hr., the solution cooled, 20 ml. H_2O added, the solution treated with C, filtered, and the filtrate neutralized gave 0.6 g. Me 1,2,3,4,9,10-hexahydro-6,7-dimethoxy-2-methyl-10-oxo-9-oxa-3,1'- diazaindeno(2',3'-2,3)-anthracene-5-carboxylate, prisms, m. 209-11° (EtOH) [picrate, plates, m. 263° (decomposition) (HOAc)]. VI (1.0 g.) refluxed 1 hr. with 0.65 g. $HONH_2.HCl$ and 1.4 g. $NaOAc.3H_2O$ in 20 ml. MeOH, the mixture filtered, the filtrate concentrated to 10 ml., diluted with 10 ml. H_2O, the mixture kept several days, and the resulting precipitate (0.15 g.) repeatedly crystallized from EtOH gave Me 6,7-dimethoxy-6'-

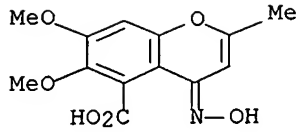
methylpyridino(4',3'- 2,3)-chromone-5-carboxylate 1'-oxide, yellow prisms, m. 266° (decomposition), giving a neg. ferric reaction, liberating iodine from HI, λ 242, 282, 326 mμ (log ε 4.08, 4.49, 4.38), characteristic infrared spectrum. N led 0.75 hr. through 3.32 g. VI in 10 ml. boiling II or MeOH containing 80 ml. 2N aqueous NaOH and the effluent gases tested gave neg. tests for carbonyl compds. The solution kept 8 hrs. at 0° deposited 0.5 g. Na salt (IX) and the filtrate from IX gave an addnl. 1.1 g. IX. IX decomposed by acids furnished 1.2 g. 2-acetyl-7-hydroxy-4,5-dimethoxyindan-1,3-dione (X), pale yellow needles, m. 157°, giving a purple-brown ferric reaction, mixed m.p. with so-called 6,7-dimethoxy-2-methylchromone-5-carboxylic acid prepared from citromycetin (XI) 157°; the 2 specimens have identical ultraviolet and infrared spectra, λ 300 mμ (log ε 4.52) [oxime, yellow needles, m. 213-14° (EtOH)]. Methylation of X with MeI and K2CO3 in Me2CO gave the di-Me ether, needles, m. 77°, giving a neg. ferric reaction. After removal of the X, the acidified hydrolyzate was distilled almost to dryness, the distillate treated with KMnO4, the excess KMnO4 destroyed with H2O2 and dilute H2SO4, and the distillate redistd., giving 1.03 ml. HOAc, identified by conversion into IV, m. 175-6°, and into AcNHPh, m. 112°. Ozonolysis of VI gave no definite results. CrO3 in 80% HOAc added dropwise at 50° during 1 hr. to VI, and the mixture kept 1 hr. at 50° and worked up gave Me 6,7-dimethoxy-6'-methyl-α-pyrano[4',3'-2,3]chromone-5-carboxylate, pale cream plates, m. 250° (decomposition), inert toward FeCl3 and [2,4-(O2N)2C6H3NHNH2]2.H2SO4 (XII). KMnO4 (9 g.) added during 2.25 hrs. to 2.5 g. VI in 200 ml. boiling Me2CO, the mixture later clarified by 100 ml. H2O and SO2, concentrated in vacuo to 120 ml., the product extracted with eight 50-ml. portions of Et2O, the extract washed with H2O, extracted with two 25-ml. portions of aqueous NaHCO3 (a separation from 30 mg. neutral nonketonic substance, prisms, m. 206°), the alkaline extract acidified with dilute HCl, the product (XIII) (1.2 g.) isolated with Et2O, the XIII extracted with C6H6, and the insol. material crystallized from aqueous II gave 0.1 g. Me 4-hydroxy-6,7-dimethoxycoumarin-5-carboxylate, cream prisms, m. 255-6° (decomposition), brown ferric reaction; the C6H6 extract allowed to stand and the product which separated recrystd. from C6H6 gave 0.7 g. 6,3,4,2-HO(MeO)2(MeO2C)C6HCO2H, m.p. and mixed m.p. (with sample prepared from XI) 147-8°, characterized by conversion into 6-ethoxy-3,4-dimethoxyphthalic anhydride, m. 195°. Powdered KMnO4 (2.0 g.) added at 0° during 5 hrs. to 2.0 g. VI in 200 ml. Me2CO, the mixture clarified with 100 ml. H2O and SO2, concentrated in vacuo below 40°, extracted with six 25-ml. portions of EtOAc, and the extract washed with H2O and two 25-ml. portions of dilute NaHCO3, and evaporated gave 0.5 g. recovered VI, m. 192° (decomposition); the alkaline extract acidified, and the resulting solid (0.55 g.) leached with 2 10-ml. portions of boiling C6H6 and crystallized from aqueous MeOH gave 3-acetoxymethyl-6,7-dimethoxy-5- carbomethoxychromone-2-carboxylic acid, needles, m. 233-4° (decomposition), giving a neg. test with FeCl3 and XII, λ 221, 314 mμ (log ε 4.39, 3.97), inflections at 235, 292 mμ (log ε 4.35, 3.89) [forming with CH2N2 the Me ester, m. 195° (MeOH)]. VI (0.95 g.) in 10 ml. CHCl3 treated with 1.05 moles BzO2H in CHCl3, the solution extracted after 1 hr. with aqueous NaHCO3, washed with H2O, dried, evaporated, and the residue fractionally crystallized from C6H6 gave Me 2-(1-hydroxyacetonyl)-6,7-dimethoxychromone-5-carboxlate, faint yellow needles, m. 202-4° (decomposition), λ 314 mμ (log ε 3.99); from the fractionation, a very small amount of a compound, m. 237-8° (decomposition) (EtOAc), was also obtained.

IT 100394-34-1

(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 100394-34-1 CAPLUS

CN 4H-1-Benzopyran-5-carboxylic acid, 6,7-dimethoxy-2-methyl-4-oxo-, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 49 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1958:25509 CAPLUS Full-text
DOCUMENT NUMBER: 52:25509
ORIGINAL REFERENCE NO.: 52:4615g-i,4616a-i,4617a-b
TITLE: Vitexin. I
AUTHOR(S): Evans, W. H.; McGookin, A.; Jurd, L.; Robertson, Alexander; Williamson, W. R. N.
CORPORATE SOURCE: Trinity Coll., Dublin, Ire.
SOURCE: Journal of the Chemical Society (1957) 3510-23
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB cf. Nakaoki, C.A. 46, 108c. Extraction of 20 kg. Vitex littoralis by the method of Perkin (J. Chemical Society 73, 1019(1898)) gave 215 g. vitexin (I), m. 264-5°, [α]20D -14.35 (c 2.414, C5H5N). I (0.2 g.) with 2 ml. Ac2O and 0.5 g. NaOAc at 100° 1 hr. gave vitexin heptaacetate (II), m. 257-8°, [α]20D -73.2° (Me2CO) (neg. ferric test). Similarly, I with 2 g. AcCl in 5 ml. C5H5N gave II. I (10 g.) in 100 ml. C5H5N and 100 ml. Ac2O was heated 2 hrs. on a steam bath, then diluted with 2N HOAc to precipitate vitexin pentaacetate (III), m. 146-7° then 247° (decomposition), [α]20D -4.43°. Addition of excess ethereal CH2N2 to III in methanol gave tetra-O-acetyldi-O-methylvitexin (IV), m. 205-6°, [α]20D -13.52°. IV with NH3 18 hrs. gave di-O-methylvitexin (V), m. 182° then 264°. V with Ac2O-C5H5N on a steam bath 2 hrs. gives IV. Methylation of 4.15 g. III with 40 g. K2CO3 and 40 ml. MeI in 100 ml. boiling Me2CO 8 hrs. gave a gum. The gum in warm C5H5N was treated with hot H2O until precipitation began then cooled to give a product (Va) having a slight ferric reaction. Remethylation of Va gave tetra-O-acetyltri-O-methylvitexin (VI), m. 202°, 212°, [α]20D -9.82°. VI with NH3 gave tri-O-methylvitexin (VII), m. 288°; tetrakis(p-nitrobenzoate), m. 176°. VII in Ac2O-C5H5N gave VI. Similarly, III with K2CO3 and EtI gave tetra-O-acetyltri-O-ethylvitexin (VIII), m. 236°. VIII with NH3 gave tri-O-ethylvitexin (IX), m. 270°. VII (11 g.), 25 g. Ag2O, 40 g. MeI, and 500 ml. Me2CO was refluxed 50 hrs., then evaporated to give a gum which was similarly remethylated 24 hrs. Evaporation gave a gum which was taken up in C6H6. Stepwise addition of petr. ether to the warm solution, then cooling separated an oil (IXa), then a solid, hexa-O-methylvitexin (X), m. 205°, [α]20D -13.45° (MeOH). Addition of the mother liquor to IXa and concentration gave more X, then penta-O-methylvitexin; m. 220° (p-nitrobenzoate, m. 277°). VII was hydrolyzed at reflux with 8% aqueous NaOH under N, then steam distilled giving a product (Xa). Acidification of the residue gave p-anisic acid (XI), m. 182° (amide, m. 164°). A portion of Xa was treated with 2,4-dinitrophenylhydrazine sulfate and the precipitate chromatographed on Al2O3 with C6H6 to give p-methoxyacetophenone 2,4-dinitrophenylhydrazone (XII), m. 256°. Extraction of remaining Xa gave p-MeOC6H4Ac; semicarbazone, m. 198°. VII was hydrolyzed with boiling saturated aqueous Ba(OH)2 under a rapid stream of N. The effluent passed into aqueous 2,4- dinitrophenylhydrazine gave XII. The alkaline residue was acidified and filtered. Extraction of the precipitate with MeOH gave XI. The filtrate was treated with BaCO3, filtered, and evaporated to give di-O-methylapovitexin (XIII), C14H16O7(OMe2), m. 126-30°, then 222° (decompose), [α]20D -4.59°; pentakis-(p-nitrobenzoate), m. 192°; pentaacetate, m. 151-2°. Aqueous HIO4

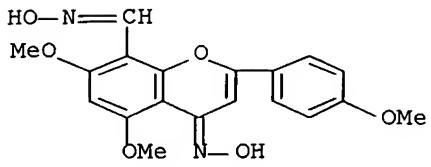
(10 ml. 6%) was added to 0.2 g. XIII in 10 ml. HOAc, the mixt agitated 5 hrs., diluted with 100 ml. H2O, then kept 16 hrs. to give 3-formyl-2-hydroxy-4,6-dimethoxyacetophenone (XIV), m. 170°; 2,4-dinitrophenylhydrazone, m. 259°. Reduction of 4 g. XIV with 10 g. Zn-Hg in 30 ml. HOAc and 8 ml. concentrated HCl 2-3 min. and dilution with H2O gave 2.5 g. 2-hydroxy-4,6-dimethoxy-3-methylacetophenone, m. 144°. Hydrolysis of IX with Ba(OH)2 gave di-O-ethylapovitexin, m. 203.5-204°; pentakis(p-nitrobenzoate), m. 175°. VII (1.5 g.), 4.5 g. Pb(OAc)4, and 25 ml. HOAc was kept at 25° 5 days, poured into H2O, extracted with CHCl3, the extract washed with NaHCO3, dilute NaOH, and H2O, then evaporated to give 8-formyl-5,7,4'- trimethoxyflavone (XV), m. 237°; 2,4-dinitrophenylhydrazone, m. 250-4°; dioxime, m. 232°. Hydrogenation of XV over Raney Ni in dioxane gave 5,7,4'-trimethoxy-8-methylflavone (XVI), m. 230°. 2-Hydroxy-4,6-dimethoxy-3-methylacetophenone (13 g.), 12 g. p-MeOC6H4COCl, and 75 ml. C5H5N was heated 3 hrs. on a steam bath, cooled, and poured into 500 ml. H2O to give 16 g. 2-p-anisoyloxy-4,6-dimethoxy-3- methylacetophenone (XVII), m. 169°. XVII (7 g.) was refluxed with 35 g. NaNH2 in 75 ml. C6H6 4 hrs. to give 4 g. 2-hydroxy-4,4',6-trimethoxy- 3-methyldibenzoylmethane (XVIII), m. 184°; monoxime, m. 188°. Cyclization of XVIII with 75% H2SO4 10 min. at room temperature gave XVI. VII (0.3 g.), 5 ml. HNO3, and 25 ml. H2O was refluxed 1.5 hrs., cooled, filtered, basified, and extracted with CHCl3 to give XV. VII (1 g.) in 150 ml. MeOH was treated with 35 ml. 0.5N HIO4, the mixture kept 10 hrs. in the dark, filtered, neutralized with Ba(OH)2, filtered, the filtrate evaporated, and the residue extracted with CHCl3 to give 0.1 g. XV. A stirred solution of 2 g. VII in 40 ml. HOAc was treated with 70 ml. 0.5N HIO4 added in 10 ml. portions at 1 hr. intervals, the mixture diluted with excess saturated aqueous NaHCO3, kept 4 days at room temperature, and filtered. Extraction of the precipitate with boiling MeOH gave 0.9 g. dehydro-O-methylsecovitexin (XIX), m. 197-8°; bis(p-nitrobenzoate), m. 159-60°. XIX (0.2 g.) in 10 ml. MeOH, 10 ml. H2O, and 2 ml. H2SO4 gave a distillate containing MeCOCHO, isolated as the 2,4-dinitrophenylosazone, m. 299-300°, and the phenylosazone, m. 145°. I (8.3 g.) in 1 l. MeOH and 300 ml. H2O was treated with 15 g. NaIO4 in 1 l. H2O, kept 20 hrs., and filtered to give 4.2 g. dehydrosecovitexin (XX), m. above 360°, [α]20D -147° (C5H5N); pentaacetate, m. 242°, [α]20D -68.25° (HOAc); pentakis(p-nitrobenzoate), m. 225°. Concentration of the filtrate from XX and extraction with Et2O gave 8-formylapigenin (XXI), m. 301°. Methylation of XXI gave XVI. Distillation of XX in the same manner as XIX also gave MeCOCHO. XX (10.1 g.), 350 ml. MeOH, and 10 ml. H2SO4 was refluxed 2.5 hrs., diluted with 1.5 l. Et2O and washed several times with aqueous NaHCO3. The Et2O was evaporated and the residue dissolved in Me2CO. The solution was concentrated until precipitation began, then cooled to precipitate 1.1 g. C18H12O6(OMe)2, m. above 360°, [α]20D 90.5°; tris(p-nitrobenzoate), m. 277°; di-Me ether (XXII), m. 247-9°, [α]20D -86.3° (MeOH). XXII gave a p-nitrobenzoate, m. 145°. The Me2CO-filtrate was diluted with EtOAc and the Me2CO evaporated On standing, 3.5 g. C18H12O6(OMe)2.H2O precipitated, m. 188-90°, [α]20D -118°; tris(p-nitrobenzoate), m. 254-5°; di-Me ether (XXIII), m. 251-2°, [α]20D -68.5°. XXIII gave a p-nitrobenzoate, m. 124-6°. XX (2.0 g.), 80 ml. MeOH, and 2 ml. H2SO4 was refluxed 2 hrs. then poured into excess BaCO3. After 0.5 hr. the filtered mixture was treated with more BaCO3, filtered, and evaporated in vacuo. The residue was extracted with warm Me2CO. The extract was evaporated and the residual oil in C5H5N treated with p-O2NC6H4COCl to give D-glyceraldehyde di-Me acetal bis(p-nitrobenzoate), m. 104-6°, [α]18D -59.6° (CHCl3). XIII (0.5 g.) in 100 ml. H2O was treated with 0.32 g. NaIO4 in 50 ml. H2O, the solution kept overnight, and the H2O removed by successive concns. and addns. of Me2CO then C6H6. After 5 days was isolated 0.34 g. dehydrodi-O- methylsecoapovitexin (XXIV), m. 158-9°, [α]23D -30.7°. Hydrolysis of 0.72 g. XXIV in 32.3 ml. MeOH and 0.82 ml. H2SO4 at reflux 0.75 hr. gave 3-acetyl-2-hydroxy-4,6-dimethoxyphenylacetaldehyde, m. 114-16°.

IT 102174-83-4

(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 102174-83-4 CAPLUS

CN 4H-1-Benzopyran-8-carboxaldehyde, 5,7-dimethoxy-2-(p-methoxyphenyl)-4-oxo-, dioxime (6CI) (CA INDEX NAME)



L9 ANSWER 50 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1958:6368 CAPLUS Full-text

DOCUMENT NUMBER: 52:6368

ORIGINAL REFERENCE NO.: 52:1151i,1152a-i,1153a-c

TITLE: Chromane. V. A new synthesis of khellin and other furo-2-methylchromones

AUTHOR(S): Dann, Otto; Illing, Gerhard

CORPORATE SOURCE: Univ. Erlangen, Germany

SOURCE: Ann. (1957), 605, 146-57

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 52:6368

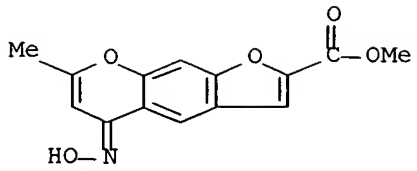
AB cf. C.A. 50, 2564c, 2566c, 2570f; 51, 6943d. The difficulties in preparing khellin (I) (2-methyl-5,8-dimethoxyfuro-2',3'; 7,6-chromone) are reviewed. 2,4-Dihydroxy-3,6-dimethoxybenzaldehyde (0.1 mole) in 250 cc. dry Me_2CO was stirred with 0.36 mole K_2CO_3 with the dropwise addition of 0.1 mole $PhCH_2Br$, refluxed 3 hrs., 0.36 mole K_2CO_3 added, and 0.11 mole $BrCH_2CO_2Me$ added dropwise, the mixture refluxed 8 hrs., filtered hot, washed with Me_2CO, evaporated, refluxed 4 hrs. with 10 g. Mg filings and 350 cc. dry MeOH, treated with ice, 2N HCl added at 0°, and stirred with 1 l. H_2O. The product, m. 102°, was debenzylated by AcOH or MeOH giving 4,7-dimethoxy-6-hydroxy-2-carbomethoxycoumarone (II) (cf. Baxter, et al., C.A. 44, 155h). II refluxed in C_6H_6 with AcCl and Mg gave 91% 6-Ac derivative of II, m. 122° which treated with anhydrous HF gave II. 6-Hydroxy-2-carbomethoxycoumarone (IIa) (0.01 mole) was stirred 2 hrs. at 100° with 0.01 mole cis-MeCCl:$CHCO_2H$ in 30 cc. H_3PO_4 and 50 g. P_2O_5, poured into ice H_2O, filtered from 0.5 g. resin, and the filtrate extracted with $CHCl_3$ giving 0.87 g. mixture (III), m. poorly 213-18°, giving in poor yield with $HONH_2.HCl$ and pyridine 2-methyl-5'-carbomethoxy- 2',3';5,6(or 2',3';7,6)-chromonoxime, $C_{14}H_{11}O_5N$, m. 205° (aqueous MeOH). III crystallized repeatedly from EtOH and then from MeOH gave 4-methyl-5'-carbomethoxyfuro-2',3';5,6(or 2',3';7,6)-coumarin (IV), $C_{14}H_{10}O_5$, λmaximum 255 and 290 mμ (log ε 4.6 and 4.19), λmin. 283 mμ (log ε 4.14), giving no oxime. IIa (0.01 mole) and 0.01 mole cis-MeCCl:$CHCO_2Me$ refluxed and stirred 8 hrs. in 100 cc. dry Me_2CO containing 0.075 mole K_2CO_3 gave Me β-(5'-carbomethoxyfuro-2',3';3,4-phenoxy)crotonate (V), m. 138° (EtOH). V (2.9 g.) in 30 cc. H_3PO_4 containing 60 g. P_2O_5 kept 1 hr. at 20° and stirred 5 hrs. at 70°, decomposed dropwise with ice H_2O, and the filtrate extracted with $CHCl_3$ gave 0.78 g. IV. 6-Hydroxycoumaran (VI) (1.36 g.) and 0.86 g. MeCH:$CHCO_2H$ in 30 cc. HF kept 2 days at 20° and shaken occasionally gave 0.8 g. 6-hydroxy-5-crotonylcoumaran, yellow, m. 121° (aqueous MeOH), which in little MeOH with 1% NaOH gave 2-methyl-4',5'-dihydrofuro- 2',3';7,6-chromanone, m. 94° (aqueous MeOH); oxime, m. 206° (EtOH). VI and trans-MeCCl:$CHCO_2H$ in HF gave 35% 6-hydroxy-5-(β-chlorocrotonyl)coumaran, yellow, m. 114°, which in 1.5% NaOH yielded 2-methyl-

4'5'-dihydrofuro-2',3';7,6-chromone (VII), m. 164° (H2O). As in the preparation of V, VI and trans-MeCCl:CHCO2Me gave 85% Me β-(4',5'-dihydrofuro-2',3';3,4-phenoxy)crotonate, m. 81° (aqueous MeOH); free acid (VIII), m. 180° (with loss of CO2). VIII with AcCl and a few drops H2SO4 stirred, freed from excess AcCl, and poured into ice H2O gave VI, m. 166-7°. Formed by methods analogous to those described were: Me β-(2,5-dimethoxy-4',5'-dihydrofuro-2',3';3,4- phenoxy)crotonate (IX), m. 134° (prepared from 4,7-dimethoxy-6-hydroxycoumaran); free acid, C14H16O6 (X), m. 190° (decomposition). 4-Methyl-4',5'-dihydrofuro-2',3';7,6-coumarin m. 167° (m. 143° when mixed with VII), λmaximum 225 and 230 mμ (log ε 4.08 and 4.18), λmin. 265 mμ (log ε 3.16). IX (2 g.) in 70 cc. absolute Et2O at -5° stirred with 2 g. MeCN and 4 g. dry ZnCl2, cooled below 0°, saturated with dry HCl, and kept 5 days gave a precipitate which was washed with Et2O and decomposed with 250 cc. H2O at 90° giving 1.8 g. 5-Ac derivative of IX (dihydrokhellinone), pale yellow, m. 105°. X (0.9 g.) with AcCl and H2SO4 gave 0.25 g. 4',5'-dihydro derivative of I, m. 151°. H2SO4 (85%) (10 cc.) at 0° added dropwise to 1 g. IX and 0.7 cc. AcCH2CO2Et and kept 48 hrs. at 20° gave a precipitate which was purified by solution in 2N NaOH and precipitation with HCl giving 1.3 g. 4-methyl-5-hydroxy-6-methoxy-4',5'- dihydrofuro-2', 3';7,8(or 7,6)-coumarin, C13H12O5, m. 270°, λmaximum 328 mμ (log ε 4.09), λmin. 278 mμ (log ε 3.12) (EtOH), λmaximum 320 mμ (log ε 4.21), λmin. 2.57 mμ (log ε,3.21) (MeOH), which with EtI and K2CO3 in dry MeOHMe2CO gave the Et ether, C15H16O5, m. 114° (H2O). Formed analogously to V was 75% Me β-(furo-2',3';3,4- phenoxy)crotonate, m. 45-6° (75% MeOH); free acid, m. 179° (decomposition), cyclized gave 2-methylfuro-2',3';5,6-chromone, m. 225° (MeOH); oxime, m. 139° (decomposition). 1,3,2,5-(HO)2(MeO)2C6H2 (68 g.) in 1 l. absolute Et2O stirred below 0° with 55 g. anhydrous ZnCl2 and 30 g. ClCH2CN, then saturated with dry HCl, stirred 24 hrs., and the resulting precipitate washed with Et2O and warmed with 0.5 l. H2O gave 2,4-dihydroxy-3,6- dimethoxy-ω-chloroacetophenone, m. 148°, which was kept 5 hrs. in 750 cc. N NaOH, cooled to 0°, and acidified with concentrated HCl giving 65 g. 4,7-dimethoxy-6-hydroxy-3-coumaranone (XI), m. 178-80° (after washing with ice H2O, and drying in vacuo), turning red in air. A similar reaction carried out with 1,4,2,6-(MeO)2(PhCH2O)2C6H2 gave the 6-PhCH2 derivative of XI, m. 123°; oxime, m. 194-5° (decomposition). The oxime (XII) of XI darkened on heating and decomposed about 198°. XII (30 g.) slurried at 40-50° with 600 cc. EtOH and 80 cc. glacial AcOH was treated gradually with 1.5 kg. 2.5% Na-Hg and concomitantly, dropwise, with enough glacial AcOH to insure an acid mixture, stirred 12 hrs. at 20°, decanted, and the residue washed with H2O. All solns. were mixed and evaporated in vacuo; the residue was refluxed 2 hrs. with 500 cc. H2O, and this solution extracted with Et2O; the dried, evaporated extract gave 14 g. 4,7-dimethoxy-6-hydroxycoumarone, b1.2 145°, n22D 1.5721, rapidly turning orange, 5.83 g. of which with 5 g. trans-MeCCl:CHCO2Me in Me2CO with K2CO3 gave a product which on attempted distillation at 3 mm. decomposed at 210°, and which, saponified (without distillation) gave 58% β-(2,5-dimethoxyfuro-2',3';3,4-phenoxy)crotonic acid, m. 189° (decomposition), 1.8 g. of which with 15 cc. AcCl and 5 drops H2SO4 after 10 days gave 0.52 g. I, m. 152-3°. 20 references.

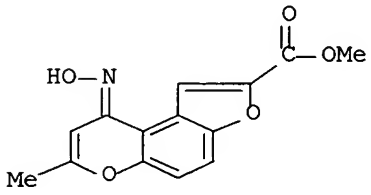
IT 107558-98-5P, 5H-Furo[3,2-g][1]benzopyran-2-carboxylic acid, 7-methyl-5-oxo-, methyl ester, oxime 110060-05-4P, 9H-Furo[2,3-f][1]benzopyran-2-carboxylic acid, 7-methyl-9-oxo-, methyl ester, oxime 116055-72-2P, 9H-Furo[2,3-f][1]benzopyran-9-one, 7-methyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 107558-98-5 CAPLUS

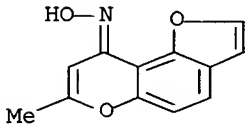
CN 5H-Furo[3,2-g][1]benzopyran-2-carboxylic acid, 7-methyl-5-oxo-, methyl ester, oxime (6CI) (CA INDEX NAME)



RN 110060-05-4 CAPLUS
CN 9H-Furo[2,3-f][1]benzopyran-2-carboxylic acid, 7-methyl-9-oxo-, methyl ester, oxime (6CI) (CA INDEX NAME)



RN 116055-72-2 CAPLUS
CN 9H-Furo[2,3-f][1]benzopyran-9-one, 7-methyl-, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 51 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1957:21784 CAPLUS Full-text
DOCUMENT NUMBER: 51:21784
ORIGINAL REFERENCE NO.: 51:4401b-h
TITLE: The constituents of Casimiroa edulis. I. The seed
AUTHOR(S): Kincl, F. A.; Romo, J.; Rosenkranz, G.; Sondheimer, Franz
CORPORATE SOURCE: Syntex S. A., Mexico D. F., Mex.
SOURCE: Journal of the Chemical Society (1956) 4163-9
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

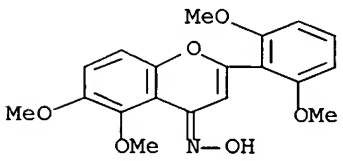
AB cf. Power and Callan, C.A. 6, 667. The dried, ground kernels were extracted twice with 400 l. hot EtOH. The combined exts. were evaporated and diluted with 50 1.4% aqueous HCl. The mixture was extracted (5 + 10 l. each) with C6H14 (A), C6H6 (B), CH2Cl2 (C), and AmOH (D). The aqueous layer was basified with aqueous NH3 and extracted similarly to give the basic exts. (E, F, G, and H), resp. A was chromatographed on 40 parts of Al2O3. Elution with 7:3 C6H6-Et2O gave β-sitosterol, m. 138-9°, [α]D -38°; acetate, m. 127-8°, [α]D -38°; benzoate, m. 145-7°, [α]D -12°. Further elution with 1:1 C6H6-Et6O gave

palmitamide, m. 103-4°. Chromatography of B and elution with 4:1 C6H6-Et6O gave zapotin, C19H18O6 (I), m. 150-1 (picrate, m. 181-2°; perchlorate, m. 204-6°; oxime, m. 240-3°); 3:1 C6H6-Et2O gave casimiroin, C12H11NO4 (II), m. 202-3° (picrate, m. 193-4°; aurichloride, m. 196-8°); 4:1 Et2O-EtOAc gave N-benzoyltyramine (III), m. 161-2° (acetate, m. 121-2°; benzoate, m. 172-3°). I (4 g.) was refluxed 1 hr. with 60 ml. Ac2O and 85 ml. aqueous HI to yield 3.1 g. of demethylzapotin, C15H10O6 (IV), m. 321-5°, green with alc. FeCl3. KOH-fusion of IV yielded salicylic acid, m. 156-8°, and resorcinol (dibenzoate, m. 116.5°). Refluxing II 20 min. in 20% aqueous HCl gave casimiroinol, C11H9NO4 (V), m. 321-3°. V with CH2N2 gave II. KOH-fusion of III gave BzOH. III with CH2N2 gave the Me ether, m. 123-4° which was oxidized with alkaline KMnO4 to give p-MeOC6H4CO2H. C yielded 9-hydroxy-4- methoxyfurano[3,2-g]benzopyran-7-one (VI), m. 223-4°; acetate, m. 181-2° benzoate, m. 203-5°. VI with CH2N2 gave isopimpinellin, C13H10O5, m. and mixed m.p. 150-1°. VI with CrO3-AcOH gave bergaptenquinone, m. 251-3° (decomposition). VI in alkaline KMnO4 gave 2,3-furandicarboxylic acid, m. 220-1°. Chromatography of the mother liquor from C and elution with C6H6 gave zapotinin, C18H16O6 (VII), m. 224-5° green with alc. FeCl3 (acetate, m. 214-16°); C6H6-CH2Cl2 gave zapoterin, C19H24O6 (VIII), m. 257-9° [α]D -51°; CH2Cl2 gave casmirolid, m. 229-31, [α]D -49°. KOH-fusion of I at 270° for 20 min. gave VII. IV with CH2N2 also gave VII. VIII kept 1 hr. with Ac2O and C5H5N at 90° gave isozapoterin, m. 284-5°. D separated β-sitosterol β-D-glucoside, m. 290-5° (decomposition); tetraacetate, m. 164-6°. Chromatography of F and elution with 9:1 C6H6-Et2O gave eduline, C17H15-NO2, m. 187-8°; picrate, m. 225-7° perchlorate, m. 250-2°. Chromatography of G and elution with C6H6 gave zapotidine, C7H9N3S, m. 96-8°; picrate, m. 195-6°. H crystallized casimiroedine, C17H24N2O5, m. 224-5°, [α]D -33°.

IT 111441-11-3
(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 111441-11-3 CAPLUS

CN Zapotin, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 52 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1957:21783 CAPLUS Full-text

DOCUMENT NUMBER: 51:21783

ORIGINAL REFERENCE NO.: 51:4401a-b

TITLE: Alkaloid studies. XIV. The structure of the cactus alkaloid pilocereine

AUTHOR(S): Djerassi, Carl; Figdor, S. K.; Bobbitt, J. M.; Markley, F. X.

CORPORATE SOURCE: Wayne Univ., Detroit, MI

SOURCE: Journal of the American Chemical Society (1956), 78, 3861-2
CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

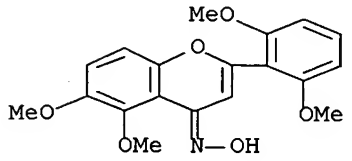
GI For diagram(s), see printed CA Issue.

AB In an abstract of this paper (C.A. 51, 1217f), the lower half of formula I should be as follows:

IT 111441-11-3
(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 111441-11-3 CAPLUS

CN Zapotin, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 53 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1956:89182 CAPLUS Full-text

DOCUMENT NUMBER: 50:89182

ORIGINAL REFERENCE NO.: 50:16759g-i,16760a

TITLE: Structure of sciadopitysin, a flavonoid from the leaves of Sciadopitys verticillata. IV. Degradation of sciadopitysin trimethyl ether in ethanolic potassium hydroxide solution

AUTHOR(S): Kawano, Nobusuke

SOURCE: Yakugaku Zasshi (1956), 76, 457-61
CODEN: YKKZAJ; ISSN: 0031-6903

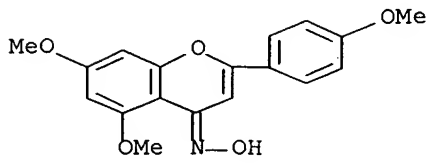
DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

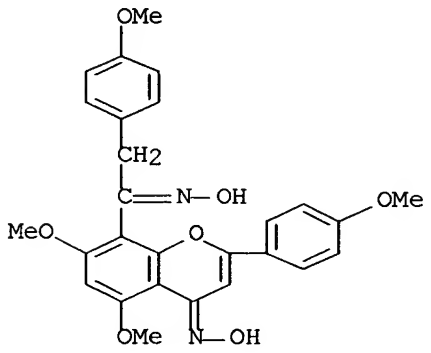
AB IIIa (1 g.) and 15 mL. 15% KOH-EtOH refluxed 3.5 h., the solution acidified with H2SO4, the precipitate filtered off and boiled 30 min. with 20 mL. 30% KOH, 80 mL. water added, the mixture extracted with Et2O, and the extract evaporated gave p-MeOC6H4Ac (semicarbazone, m. 198-9°); the mother liquor extracted with Et2O and saturated with CO2 and the precipitate filtered off and recrystd. from EtOH gave 50 mg. putative 5,2,4,6-Ac(MeO)2(HO)C6HCOCH2C6H4OH-x (VII), plates, C19H20O6, m. 226-7°; the mother liquor from VII extracted with Et2O gave 10 mg. 6,2,4-HO(MeO)2C6H2Ac, m. 79-80°; this mother liquor evaporated, the residue acidified with H2SO4, the precipitate (VIIa) filtered off, and the filtrate extracted with Et2O gave 10 g. anisic acid, m. 179-81°. VIIa and 5 mL. Me2CO concentrated, 2 mL. MeOH added, and the mixture allowed to stand gave 10 mg. putative 6,2,4-HO(MeO)2C6H2COCH2C6H3(OMe)CO2H-x,x' (VIII), columns, m. 293-4°. The mother liquor from VIII evaporated to dryness, the residue extracted with CCl4 and recrystd. from dilute EtOH gave 6,2,4-HO(MeO)2C6H2CO2H, m. 156-8° (decomposition). IV di-Me ether (400 mg.) and 6 mL. 15% KOH-EtOH refluxed 40 min. and the product treated as in the preparation of VII gave 20 mg. p-MeOC6H4Ac, 15 mg. VII, and 15 mg. putative 6,5,2,4-HO(HO2C)(MeO)2C6HCOCH2C6H4OMe-x (IX), m. 217-18° (decomposition), and 8 mg. anisic acid. VII forms a monoxime, C19H21O6N, m. 230-2°; Ac derivative of VII, C21H22O7, m. 169° (dioxime, C21H24O7N2, m. 207°). Me ester of VIII, C19H20O7, m. 202°; mono-Me ether (VIIIa) of VIII, C19H20O7, m. 203° (decomposition); VIIIa oxime, C19H21O7N, m. 231°. Ac derivative of VIII, C20H20O8.H2O, m. 119-20° (oxime, C20H21O8N, m. 242°). It is suggested that I is 5-hydroxy-2-[5-hydroxy-7-methoxy-2-(4-methoxyphenyl)-4-oxo-4H-1-benzopyran-8-yl]-3-(4-hydroxyphenyl)-7-methoxychromone.

IT 872302-08-4P, Flavone, 4',5,7-trimethoxy-, oxime
RL: PREP (Preparation)
(preparation of)

RN 872302-08-4 CAPLUS
CN Flavone, 4',5,7-trimethoxy-, oxime (5CI) (CA INDEX NAME)



L9 ANSWER 54 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1956:89181 CAPLUS Full-text
DOCUMENT NUMBER: 50:89181
ORIGINAL REFERENCE NO.: 50:16759f-g
TITLE: Structure of sciadopitysin, a flavonoid from the leaves of Sciadopitys verticillata. III. The structure of oxoflavone and carboxyflavone
AUTHOR(S): Kariyone, Tatsuo; Kawano, Nobusuke
SOURCE: Yakugaku Zasshi (1956), 76, 453-6
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB IV and V were each shown to have 2 MeO groups and 2 phenolic HO groups and were assumed to have the skeleton of acacetin 7-Me ether (VI) with an acyl group as the side-chain. IV is VI where the acyl is COCH2C6H4OH and V is IV bearing a group on the VI moiety.
IT 874530-27-5P, Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime
RL: PREP (Preparation)
(preparation of)
RN 874530-27-5 CAPLUS
CN Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime (5CI) (CA INDEX NAME)



L9 ANSWER 55 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1956:89180 CAPLUS Full-text
DOCUMENT NUMBER: 50:89180
ORIGINAL REFERENCE NO.: 50:16759e-f

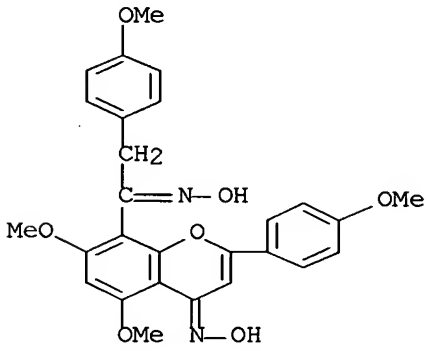
TITLE: Structure of sciadopitysin, a flavonoid from the leaves of Sciadopitys verticillata. II. Degradation of sciadopitysin
AUTHOR(S): Kariyone, Tatsuo; Kawano, Nobusuke
SOURCE: Yakugaku Zasshi (1956), 76, 451-2
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB Decomposition of I by boiling 1.5 hrs. with 20% aqueous KOH afforded an oxoflavone (IV), C25H20O7, yellow columns, m. 241-2°, a carboxyflavone (V), C26H20O9, yellow, m. 311° (decomposition), anisic acid, p-MeOC6H4Ac, and 4,2,6-MeO-(HO)2C6H2Ac; the yield of these products varied with the concentration of KOH and duration of boiling.

IT 874530-27-5P, Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime
RL: PREP (Preparation)
(preparation of)

RN 874530-27-5 CAPLUS

CN Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime (5CI) (CA INDEX NAME)



L9 ANSWER 56 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1956:89179 CAPLUS Full-text
DOCUMENT NUMBER: 50:89179
ORIGINAL REFERENCE NO.: 50:16759d-f
TITLE: Structure of sciadopitysin, a flavonoid from the leaves of Sciadopitys verticillata. I
AUTHOR(S): Kariyone, Tatsuo; Kawano, Nobusuke
SOURCE: Yakugaku Zasshi (1956), 76, 448-50
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

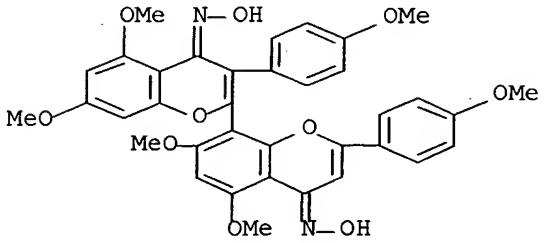
AB Air-dried leaves (3 kg.) in 20 l. CHCl:CCl2 refluxed 3 hrs., the extract concentrated to 3 l., allowed to stand overnight, the waxy precipitate warmed up, and the insol. residue filtered off gave 9 g. sciadopitysin (I), yellow columns, m. 285-6° (from C5H5NEtOH); triacetate, prisms, m. 264°; tri-Me ether (IIIa), m. 214-15° (oxime, C36H32O10N2, columns, m. 248-9°). I (0.1 g.) in 40 ml. Me2CO treated with 0.5 g. each of K2CO3 and MeI and the mixture refluxed 40 min. and concentrated gave a I mono-Me ether (II), m. 282°, identical with ginkgetin di-Me ether (III) by mixed m.p. II gives a diacetate, m. 228°, identical with the diacetate of III by mixed m.p. Demethylation of I with HI gave C30H18O10.5H2O, m. above 360°, and acetylation of this substance gave a

product, m. 240°, identical with that prepared by treating ginkgetin in a similar way.

IT 854209-81-7P, [2,8'-Bi-4H-1-benzopyran]-4,4'-dione, 5,5',7,7'-tetramethoxy-2',3-bis(p-methoxyphenyl)-, dioxime
874530-27-5P, Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime
RL: PREP (Preparation)
(preparation of)

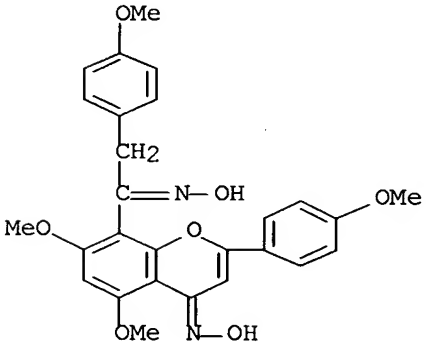
RN 854209-81-7 CAPLUS

CN [2,8'-Bi-4H-1-benzopyran]-4,4'-dione, 5,5',7,7'-tetramethoxy-2',3-bis(p-methoxyphenyl)-, dioxime (5CI) (CA INDEX NAME)



RN 874530-27-5 CAPLUS

CN Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime (5CI) (CA INDEX NAME)



L9 ANSWER 57 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:	1956:12315 CAPLUS Full-text
DOCUMENT NUMBER:	50:12315
ORIGINAL REFERENCE NO.:	50:2570f-i,2571a-i,2572a-i,2573a-f
TITLE:	Synthesis of chromanones, chromans, and 2-methylchromones with hydrofluoric acid
AUTHOR(S):	Dann, Otto; Volz, Gerda; Huber, Otto
CORPORATE SOURCE:	Univ. Erlangen, Germany
SOURCE:	Ann. (1954), 587, 16-37
DOCUMENT TYPE:	Journal
LANGUAGE:	Unavailable

AB A number of substances related structurally to certain portions of the rotenone (I) mol. have been synthesized and tested as insect and as fish

poisons. Action of HF on mixts. of phenols and acrylic acids (or on Ph acrylates) gives chromanones, which are reduced by the Clemmensen method to the corresponding chromans; use of β-chloropropionic acid (II) gives substances unsubstituted in the 2-position. Chromones are similarly prepared employing cis-β-chlorocrotonic acid (III). Ph esters (except those of II) are prepared by the procedure of Spasov (C.A. 36, 7010.2). p-Cresol (IV) crotonate (100 g., prepared in 90% yield, b15 133° nD23 1.523) is heated in a cast steel bomb with 100 mL. com. anhydrous HF for 3 h. at 100°; pouring the reaction product into ice H2O gives a brown oil which soon solidifies and is then taken up in min. hot EtOH, poured into 1.5 l. 1.5% aqueous NaOH, and shaken overnight to give 60% 2,6-dimethylchromanone (V), m. 52-4° (from petr. ether), which turns deep yellow on treatment with concentrated H2SO4. Similar treatment of a mixture of IV and crotonic acid (VI) (heated with HF 2 h. at 100°) gives 82% V, b11 135-7°. V (8 g.) in 80 mL. HOAc mixed with 200 g. amalgamated Zn dust and 300 mL. 18% HCl and let stand 24 h. at room temperature, 80 mL. 12% HCl added, the mixture refluxed 6-7 h., cooled, and extracted with Et2O, and the extract distilled gives 2,6-dimethylchroman (VII), b11 115-25° nD16 1.531. Resorcinol (VIII) dicrotonate (in HF at 18° for 3 h.) gives 60% 3-crotonoyloxy-4-crotonoylphenol (IX), deep yellow needles from C6H6, m. 138° FeCl3 test (in MeOH) deep violet; on standing overnight, the deep orange solution of IX in 1.5% NaOH lightens in color and on acidification yields 72% 7-hydroxy-2-methylchromanone (X), m. 175-6° (from C6H6). VIII and VI (with HF, 2 h. at 100°, followed by treatment with 1.5% NaOH) give X in 84% yield. The action of Me2SO4 and alkali on X (2.5 g.) gives 0.6 g. 7-methoxy-2-methylchromanone (XI), m. 74-7°. Reduction of + with amalgamated Zn and HCl gives 68% 7-hydroxy-2-methylchroman (XII), b0.5 120-5°, m. 67-8°, pale yellow with concentrated H2SO4. XII is converted by Me2SO4 and alkali to 7-methoxy-2-methylchroman (XIII) (1.5 g. from 2 g.), b12 130-50°, nD13 1.539. Heating p-cresol β-chloropropionate (36 g., prepared in 40% yield, b12 145-50°) in HF for 3 h. at 55-60° and distillation of the crude product (b11 145-55°) gives 16 g. alkali-soluble material which is refluxed 20 h. in 20% aqueous Na2CO3 to give 2,5-dimethylcoumaranone, b11 140-50°, m. 52-4° (from petr. ether), FeCl3 test neg.; oxime, m. 129°; semicarbazone, m. 191°. IV and II (20 g. each) heated 5 h. in HF at 50° gives 11 g. 2-(β-chloropropionyl)-p-cresol, yellow, m. 60-2° (from MeOH), which gives 52% 6-methylchromanone (XIV), b0.5 100-20°, m. 36° (from petr. ether), on treatment with 1.5% aqueous NaOH. Heating II and VIII in HF for 1.5 h. at 50° gives 33% 4-(β- chloropropionyl)resorcinol yellow, m. 96° (from petr. ether-C6H6), converted with 1.5% NaOH to 81% 7-hydroxychromanone, m. 146° (from EtOAc). Action of alkali on a mixture of o-cresol and II gives a 26% yield of crude β-(2-methylphenoxy)propionic acid, m. 92-3°, heating of which with HF for 5 h. at 55-6° gives 64% 8-methylchromanone (XV) (distilled in vacuo, nD22 1.572); reduction of 3 g. XV with Zn-HCl gives 0.4 g. 8-methylchroman (XVI), nD20 1.541. β-(2,5- Dimethylphenoxy)propionic acid is similarly prepared (yield 16%), needles from 50% aqueous HOAc, m. 108-10°, and converted to 5,8-dimethylchromanone (XVIA, 60%), pale yellow, b1.5 110-30° nD20 1.567; oxime, m. 116-17° (from MeOH); with 3 g. XVIA with Zn-HCl gives 0.8 g. 5,8-dimethylchroman (XVII), b5 90-110°, nD20 1.543. Similarly, 3.5 g. β-(2,3-dimethylphenoxy)propionic acid (prepared in 25% yield) gives 1.6 g. reddish 7,8-dimethylchromanone (XVIII), b3 120-40°, m. 47-8.5° [oxime, m. 165-6° (from petr. ether-C6H6)]; 1.6 g. XVIII gives 0.4 g. 7,8-dimethylchroman (XIX), b2 120-40°, nD23 1.541. HF converts β-(2,3,5- trimethylphenoxy)propionic acid (m. 118-20°, prepared in 10% yield) to 5,7,8-trimethylchromanone (XX, yield 76%), b2 100-20° (congeals to waxy solid), orange in concentrated H2SO4 [oxime, m. 142-3° (from petr. ether-C6H6)], reduced to 59% 5,7,8-trimethylchroman (XXI), b4 100-20°, m. 53° colorless in concentrated H2SO4. Heating 5 g. m-cresol crotonate (b12 135-7°, nD14 1.522, m. about 20°, prepared in 70% yield) in HF for 3 h. at 100° gives 3 g. 2,7-dimethylchromanone (XXII), b12 150-60°, nD16 1.557, citron-yellow in

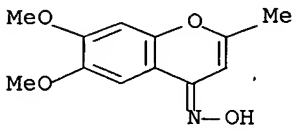
concentrated H_2SO_4 [oxime, m. 140° (from MeOH)], reduced (0.4 g. from 1.5 g.) to 2,7-dimethylchroman (XXIII), b0.5 100-10°. 3,4-Dimethylphenol (12.2 g.) and 8.6 g. VI in HF (2 h. at 100°) give 12 g. 2,6,7-trimethylchromanone (XXIV), m. 90-1° [oxime, m. 176-7° (from petr. ether-C_6H_6)]; reduction of 5 g. XXIV gives 2 g. 2,6,7-trimethylchroman (XXV), nD20 1.526, which solidifies on standing. Heated 3 h. at 100° in HF, 10 g. 3,5-dimethylphenol crotonate (b12 138-40°, prepared in 85% yield) gives 4 g. yellow 2,5,7-trimethylchromanone (XXVI), m. 65°, citron-yellow in concentrated H_2SO_4 [oxime, m. 140° (from MeOH)]; on reduction, 2.5 g. XXVI yields 1.5 g. 2,5,7-trimethylchroman (XXVII),nD22 1.528. 2,6,8- Trimethylchromanone (XXVIII), brownish needles from aqueous MeOH, m. 59-60° [oxime, m. 124-5° (from petr. ether-C_6H_6)], is obtained in 63% yield by heating 2,4-dimethylphenol with VI in HF for 2 h. at 100°; XXVIII is reduced to 43% 2,6,8-trimethylchroman (XXIX), m. 44-5°. Similarly, 6.8 g. 2,3,5-trimethylphenol (XXX) and 4.3 g. VI condense to give 5.8 g. 2,5,7,8-tetramethylchromanone (XXXI), b4 140-60°, m. 47-8° [oxime, m. 151-2° (from petr. ether-C_6H_6)]. Reduction (with ZnHCl) of 2.5 g. XXXI gives 0.9 g. 2,5,7,8-tetramethylchroman (XXXII), m. 46-8° (from aqueous MeOH). Hydroquinone dicrotonate (recrystd. from MeOH, m. 112-14°, difficultly soluble in C_6H_6) is the precursor of yellow 6-hydroxy-2- methylchromanone, m. 152-3° (from C_6H_6), orange in concentrated H_2SO_4, converted by treatment with Me_2SO_4 and alkali (but not with CH_2N_2) to 6-methoxy-2-methylchromanone (XXXIII), m. 65-7° (from C_6H_6), insol. in alkali, $FeCl_3$ test neg.; Zn-HCl reduction of 3 g. XXXIII gives 1.5 g. 6-methoxy-2-methylchroman (XXXIV), nD25 1.529. Heating 5 g. 2,4-dihydroxytoluene (XXXV) with 3.5 g. VI in HF for 2 h. at 100° gives 6 g. crude brown 7-hydroxy-2,6-dimethylchromanone (XXXVI), m. 194-6° after precipitation from EtOAc by addition of petr. ether; oxime, m. 172-4° (from EtOAc diluted with petr. ether). Action of Me_2SO_4 and alkali on XXXVI gives 7-methoxy-2,6-dimethylchromanone (XXXVII), m. 116-17°. Reduction of 6 g. XXXVI with Zn-HCl gives 2.5 g. 7-hydroxy-2,6-dimethylchroman (XXXVIII), m. 126-7° (from HOAc diluted with H_2O), which is converted to 7-methoxy-2,6-dimethylchroman (XXXIX), m. 53-4° (from HOAc diluted with H_2O), by treatment with Me_2SO_4-alkali. Similarly, 5 g. 2,6-dihydroxytoluene (XL) yields 7.5 g. crude 7-hydroxy-2,8-dimethylchromanone, m. 175-6° after precipitation from EtOAc with petr. ether; oxime, m. 194-6°, purified in the same way), which is methylated (with MeI and K_2CO_3 in acetone) to 7-methoxy-2,8-dimethylchromanone (XLI), m. 80-1° (petr. ether); and 6 g. reduced (with Zn-HCl) to 2.3 g. crude 7-hydroxy-2,8-dimethylchroman (XLII), needles from petr. ether, m. 71-3°, which is converted by the action of Me_2SO_4 and alkali to 7-methoxy-2,8-dimethylchroman (XL-III), b0.5 95-100°, nD18 1.539. Treating 5 g. 3,4-dimethoxyphenol crotonate (b0.8 149-50°, m. 35-40°) with HF at room temperature for 6 h., followed by treatment with 1.5% aqueous NaOH, gives 3.2 g. crude 6,7-dimethoxy-2-methylchromanone (XLIV), m. 117-18° (from petr. ether), deep red in concentrated H_2SO_4 [oxime, m. 172-4° (from MeOH on addition of H_2O)]; reduction of 2.7 g. XLIV gives 1.3 distilled 6,7-dimethoxy-2- methylchroman (XLV), nD20 1.545, which solidifies, m. 47-8°. Similarly, 5 g. trimethylhydroquinone dicrotonate (yellow, m. 124° from petr. ether) yields (after 3 h. at 100° in HF) 1 g. 6-hydroxy-2,5,7,8-tetramethylchromanone (XLVI), yellow needles from C_6H_6, m. 111-13° orange in concentrated H_2SO_4; oxime, needles from petr. ether-C_6H_6, m. 151-3°. XLVI (5 g. crude) is also obtained by heating 7.8 g. trimethylhydroquinone and 4.3 g. VI in HF for 2.5 h. at 100°. Methylation (Me_2SO_4-alkali) of 0.6 g. XLVI yields 0.2 g. 6-methoxy-2,5,7,8-tetramethylchromanone (XLVII), needles from MeOH, m. 81-2°, Zn-HCl reduction of which gives 6-methoxy-2,5,7,8- tetramethylchroman (XLVIII), b0.2 140-60°, which congeals to a yellow solid, m. 53-4°. Although reaction with CH_2N_2 or with MeI and K_2CO_3 in acetone was unavailing, prolonged treatment of dl-α-tocopherol with Me_2SO_4 and alkali gave the corresponding Me ether (XLIX), yellow oil, b0.005 200°, nD20.5 1.4995. p-Chlorophenol crotonate (b13 150-2°, nD18 1.535, prepared in 83% yield) (10 g.) in HF at 100° for 3 h. gives 2 g. 6-chloro-2-methylchromanone (L), m. 98-9° (from petr. ether); oxime, needles, m. 147° (from MeOH). IV and 1- cyclohexenecarboxylic acid

(LI) in HF for 2 h. at 100° give 6-methyl-2,3-cyclohexanochromanone (6-Methyl-2,3-tetramethylenechromanone) (LII), m. 82.5-5° (from aqueous MeOH); oxime, m. 168-70° (from MeOH). Reduction of 0.9 g. LII gives 0.3 g. 6-methyl-2,3-cyclohexanochroman (LIII), which solidifies after distillation Similarly, 6 g. XXXVI and 6.1 g. LI give 10 g. crude 7-hydroxy-6-methyl-2,3-cyclohexanochromanone, converted by treatment with Me2SO4 and 20% aqueous KOH to 2.2 g. yellow 7-methoxy-6-methyl-2,3-cyclohexanochromanone (LIV), m. 144-5° (from MeOH); oxime, m. 189-90° (from petr. ether-C6H6). XXX (6.8 g.) similarly yields 10 g. crude 5,7,8-trimethyl-2,3-cyclohexanochromanone (LV), distillation of which (b4 140-70°) gives a solid m. body temperature [oxime, m. 198-200° (from petr. ether-C6H6)]; reduction of 6 g. crude LV gives 1.2 g. 5,7,8-trimethyl-2,3-cyclohexanochroman (LVI), m. 66-8°. In the same way, 8.1 g. VIII and 10 g. LI yield 15 g. crude yellow 7-hydroxy-2,3-cyclohexanochromanone, treatment of which with Me2SO4-20% KOH gives 7-methoxy-2,3-cyclohexanochromanone (LVII), needles from MeOH, m. 114-15°; oxime, m. 174-5° (from petr. ether-C6H6). LVII is reduced to 7-methoxy-2,3-cyclohexanochroman (LVIII), a wax melting near body temperature XII is converted to the corresponding crotonate, b0.4 150-80°, which solidifies and is recrystd. from petr. ether, needles, m. 76-8° (yield 66% from XII); treatment of 4 g. of this with HF at 100° for 3 h. followed by chromatog. on Al2O3 and distillation of the crude product, gives a viscous red-yellow oil, b0.5 180-90°, which solidifies and is recrystd. from petr. ether to give 0.8 g. 2-methylchromanono[6,7:α,β]-γ-pyran-α'- methyl-α',β'-dihydride, C14H16O3, (LIX), m. 82-5°; oxime, m. 151-3° (from petr. ether-C6H6). Distillation of the crude product obtained by treatment of 10 g. β-naphthol crotonate (b12 197-8°, m. 46.5-8°, prepared in 76% yield) with HF at room temperature for 3 h. gives a solid recrystd. from petr. ether to yield 2 g. violet-tinged 2-methyl-5,6-benzochromanone (LX), m. 73-4°; oxime, m. 200-4° (from petr. ether-C6H6). LX is reduced to 2-methyl-5,6-benzochroman (LXI), b4 120-40°, m. 88-90°. p-Cresol cis-β-chlorocrotonate (b1 136-8° nD20 1.532) is treated with HF at 100° for 3 h. and the crude product run through a column of Al2O3 to give 70% 2,6-dimethylchromone, yellow needles from petr. ether, m. 98-100°, which condenses with piperonal in the presence of NaOMe to give yellow 6-methyl-2-(piperonylidenemethyl)chromone , m. 193-5° from EtOAc. VIII and III (m. 61°) react in HF at 18° (8 h.) to give 65% 4-(cis-β-chlorocrotonyl)resorcinol, yellow needles from petr. ether-C6H6, m. 114-16°, Beilstein and FeCl3 tests pos.; cyclization is effected with 1.5% aqueous NaOH to give 62% 7-hydroxy-2-methylchromone, m. 250° from EtOH. Similarly, 5 g. each of XXXVI and III (after 1.5 days in HF at room temperature and treatment of the crude product with 1.5% NaOH overnight) gives 4 g. 7-hydroxy-2,6-dimethylchromone, pale rose crystals from MeOH, m. 259-60°, which is treated with MeI and K2SO3 in acetone to give pale yellow 7-methoxy-2,6-dimethylchromone, recrystd. from EtOAc and sublimed at 150-70°/0.4 mm., m. 129-30°; oxime, m. 225-6° (from MeOH). Similarly, XL and III (20 h. in HF at room temperature) give 72% 7-hydroxy-2,8-dimethylchromone (LXII), pale rose needles from MeOH, m. 255-60°, difficultly soluble in Et2O or acetone; oxime, yellow, m. 178-9° (from MeOH on addition of H2O). Treatment of 1.5 g. LXII with MeI and K2CO3 gives 1.0 g. 7-methoxy-2,8-dimethylchromone, yellow needles from EtOAc, m. 141-2°. Similarly, distillation (b0.6 190-200°) of the crude product from 3,4-dimethoxyphenol and III, followed by recrystn. from MeOH, gives 0.5 g. 6,7-dimethoxy-2- methylchromone, m. 146-7°; oxime, m. 205-6° (from MeOH). Reaction of 3.8 g. XII with 3 g. III in HF at room temperature for 24 h. and treatment of the crude product with 1.5% NaOH gives 1.2 g. 2,6'-dimethyl-γ-pyrono[2',3':6,7]chroman, needles from MeOH, m. 130°, the "linear" structure of which is established by comparison of its UV spectrum (in MeOH) with those of 2,6',8-trimethyl-γ- pyrono[2',3':6,7]chroman (LXIII) and 2,6,6'-trimethyl-γ-pyrono[2',3':7,8]chroman (LXIV). LXIII(needles, m. 196-7° 0.9 g.) is prepared from 5 g. XLII and 3.5 g. III; while 2 g. XXXVIII and 1.3 g. III yield 0.2 g. yellow LXIV, m. 115-16° from petr. ether. In a fly (Musca domestica) killing

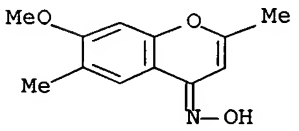
test similar to that of Wagner-Jauregg (C.A. 43, 2945a), XI, XXIV, XXVI, XXVIII, XXXI, XXXIII, XXXVII, XLI, XLIV, XLV, XLVII, XLIX, L, LII, LIV, LV, LVI, LVII, LVIII, LIX, LX, LXI, and 7-methoxy-6-methyl-2,3-cyclohexanochroman are inactive; active (like I, at 0.1 g.) are chroman itself, 6-chloro-2-methylchroman, and dihydrodeoxyrotenone; active at 0.01 g. are unsubstituted chromanone, 6-methylchroman, V, VII, XIV, XV, XVI, XVIA, XVII, XVIII, XIX, XX, XXII, XXIII, XXV, XXVII, XXIX, XXXIV, XLIII, XLVIII, and LIII; active at 0.001 g. are XIII, XXI, XXXII, and XXXIX. These activities are to be compared with that of γ-hexachlorocyclohexane (LXV), which is active in the same test at 10^{-5} g. In the test of Spath and Kuffner (C.A. 31,761.3) (against Lebistus reticulatus), the fish toxicity of V, XI, and XIII at 0.1 g. is equivalent to that of LXV at 10^{-3} g. or to that of I at 10^{-4} g.

IT 854845-38-8P, Chromone, 6,7-dimethoxy-2-methyl-, oxime
854846-08-5P, Chromone, 7-methoxy-2,6-dimethyl-, oxime
854846-33-6P, Chromone, 7-hydroxy-2,8-dimethyl-, oxime
RL: PREP (Preparation)
(preparation of)

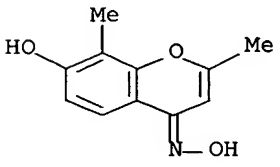
RN 854845-38-8 CAPLUS
CN Chromone, 6,7-dimethoxy-2-methyl-, oxime (5CI) (CA INDEX NAME)



RN 854846-08-5 CAPLUS
CN Chromone, 7-methoxy-2,6-dimethyl-, oxime (5CI) (CA INDEX NAME)



RN 854846-33-6 CAPLUS
CN Chromone, 7-hydroxy-2,8-dimethyl-, oxime (5CI) (CA INDEX NAME)



L9 ANSWER 58 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1956:4711 CAPLUS Full-text
DOCUMENT NUMBER: 50:4711
ORIGINAL REFERENCE NO.: 50:978a-i

TITLE: Synthesis of nuclear-substituted flavonoids and allied compounds. V. Structure of the flavone formed by degradation from ginkgetin. 2. Syntheses of 8-(2-anisoyl-ethyl)4',5,7-trihydroxyflavone methyl ethers

AUTHOR(S): Nakazawa, Koichi; Matsuura, Shin

SOURCE: Yakugaku Zasshi (1955), 75, 68-71
CODEN: YKKZAJ; ISSN: 0031-6903

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

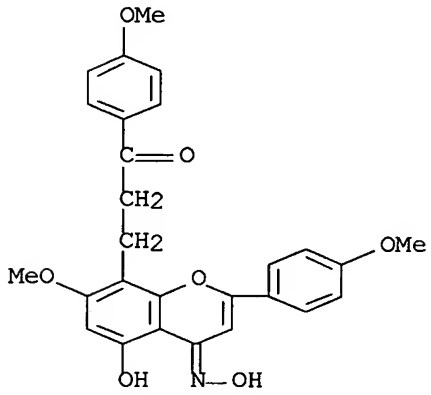
AB cf. C.A. 49, 1714e. EtOH (1 mL.), 5 mL. dioxane, and 46 mg. Na treated with 0.44 g. p-MeOC6H4COCH2CO2Et and 0.53 g. 8-chloromethyl-7- methoxyacacetin, heated 30 min. on a water bath, acidified with AcOH, diluted with water, the upper solution decanted, the precipitate in the lower layer allowed to stand 6 h. with 5 mL. each of dioxane and 10% KOH, the product diluted with water, and the precipitate filtered and recrystd. from AcOEt gave 0.4 g. 8-(2-anisoyl-ethyl)-5-hydroxy-4',7-dimethoxyflavone (I), yellow needles, m. 198°; the filtrate acidified with AcOH, and the precipitate filtered and recrystd. from AcOEt gave 0.17 g. I; 100 mg., I and 50 mg. each of NH2OH.HCl and AcONa in 2 mL. C5-H5N heated 3 h. at 110° gave 50 mg. I oxime, needles, m. 225°; 0.22 g. I and 4 mL. Me2SO4 treated with 40% KOH portionwise yielded 0.12 g. 8-(2-anisoylethyl)-4',5,7- trimethoxyflavone (II), granules, m. 152°. 2,4,6-HO(MeO)2C6H2CH2CH2CO2H (IIIa) (3.4 g.), 20 mL. MeCN, and 80 mL. dry Et2O treated with 20 g. ZnCl2, dry HCl gas passed in, the resulting solid kept 10 days in a sealed container, the Et2O removed, the residue taken up in water, the solution made weakly acid with NH4OH, washed with Et2O, boiled 30 min. and the product recrystd. from dilute AcOH gave 2.1 g. 2,4,6,3-HO(MeO)2(HO2CCH2CH2)C6HAc (III), needles, m. 179°; methylation of 0.3 g. III with 0.2 g. each of Me2SO4 and K2CO3 in 20 mL. Me2CO by refluxing 1.5 h., evaporating the solution to dryness, and recrystg. the residue from ligroine gave 0.2 g. Me ester (IV) of III, prisms, m. 147°; 0.56 g. IV in 10 mL. C5H5N heated 2 h. at 110° with 2 g. p-MeOC6H4COCl, cooled, allowed to stand 30 min. with EtOH, the solvent removed in vacuo, 20% HCl added, and the product filtered and washed with water and 10% K2CO3, gave 0.5 g. p-methoxybenzoate (V) of IV, needles, m. 121°; 0.42 g. V, 0.18 g. NaNH2, and 10 mL. xylene heated 30 min. at 110°, the product filtered hot, washed with C6H6 and Et2O, taken up in water, and CO2 passed in yielded 0.15 g. 2,3,4,6-HO(p-MeOC6H4COCH2CO)(MeO)2C6HCH2CH2CONH2(VI), yellow; the filtrate acidified with AcOH gave 30 mg. 2,3,4,6-HO(p-MeO C6H4 CO CH2CO)(MeO)2C6HCH2CH2CO2H (VII). VI (50 mg.) in 3 mL. AcOH and 1 mL. concentrated H2SO4 heated 5 min. at 100° and the product diluted with water gave 45 mg. 8-(2-carbamoylethyl)-4',5,7-trimethoxyflavone (VIII), needles, m. 282°. Cyclization of 30 mg. VII as above yielded 25 mg. 8-HO2CCH2CH2 analog (IX) of VIII, columns, m. 259°; or saponification of 100 mg. VIII in 3 g. AcOHH2SO4-H2O (2:2:1) 1.5 h. at 110° and dilution with water gave 70 mg. IX, m. 259°. IX (100 mg.) 2 drops PhOMe, and 2 g. (HPO3)n (n = 2.5) heated 30 min. at 100°, water added, the volatile substances driven off by passing in steam, and the residue recrystd. from CCl4 gave 6 mg. 8-(p-MeOC6H4COCH2CH2) analog of VIII, granules, m. 143°. Methylating 0.5 g. IIIa in 2 mL. each of Me2SO4 and MeOH in a strongly alkaline solution in 40% NaOH, acidifying the solution with HCl, and recrystg. the product from ligroine gave 0.4 g. 2,4,6-(MeO)3C6H2CH2CH2CO2H (X), needles, m. 140°; methylating 0.5 g. III as above, acidifying, and recrystg. the product from ligroine gave 0.45 g. 3,2,4,6-HO2CCH2CH2(MeO)3C6HAc (XI), needles, m. 116°. X (1.2 g.), 0.5 g. PhOMe, and 15 g. (HPO3)n heated 30 min. at 100°, the product decomposed with water, steam passed in, and the residue recrystd. from ligroine gave 0.45 g. 2,1,3,5-(p-MeOC6H4COCH2CH2) C6H2(OMe)3 (XII), leaves, m. 118 °; or 0.6 g. XI, 0.3 g. PhOMe, and 10 g. (HPO3)n treated as above gave XII. IIIa (0.4 g.), 0.2 g. AcOH, and 5 g. (HPO3)n heated 20 min. at 100° for acetylation, the product decomposed with water, and the

precipitate washed with water and recrystd. from MeOH gave 0.3 g. of the lactone, 3,5-(MeO)2C6H2.CH2.CH2.CO.O, needles, m. 105°.

IT 859441-00-2P, Flavone, 8-(2-p-anisoylethyl)-5-hydroxy-4',7-dimethoxy-, oxime
RL: PREP (Preparation)
(preparation of)

RN 859441-00-2 CAPLUS

CN Flavone, 8-(2-p-anisoylethyl)-5-hydroxy-4',7-dimethoxy-, oxime (5CI) (CA INDEX NAME)



L9 ANSWER 59 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:	1954:46252 CAPLUS Full-text
DOCUMENT NUMBER:	48:46252
ORIGINAL REFERENCE NO.:	48:8226a-d
TITLE:	Synthesis of biologically active new chromone derivatives
AUTHOR(S):	Vargha, L.; Rados, M.
CORPORATE SOURCE:	Pharm. Research Inst., Budapest
SOURCE:	Acta Chimica Academiae Scientiarum Hungaricae (1953), 3, 223-9 CODEN: ACASA2; ISSN: 0001-5407
DOCUMENT TYPE:	Journal
LANGUAGE:	German

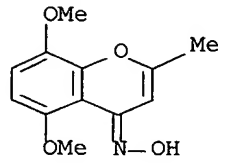
AB 2,3,6-HO(MeO)2C6H2COCHAc (I) obtained in 65% yield by treating 4.6 g. Na powder with 11 g. 2,3,6-HO(MeO)2C6H2Ac (II), 150 ml. absolute EtOH, and 6.4 g. absolute MeOH, m. 112-14° (from alc.). I (4.8 g.) in 50 ml. absolute EtOH treated with 2 ml. concentrated HCl and the product purified in vacuo gives 4 g. of a labile oxonium salt (III), m. 158-60°; III (4 g.) heated 15 min. in 150 ml. dioxane gives 3.5 g. 2-methyl-5,8-dimethoxychromone (IV), m. 129-30°; oxime, m. 107-8.5°. 2,3,4- HO(MeO)2C6H2CH2COCO2Et (V), obtained in 26 g. yield by treating 19.6 g. II and 43.8 g. (CO2Et)2 with 6.9 g. Na in 300 ml. absolute EtOH and triturating the Na salt with 10% HOAc, m. 85-7° (from H2O). 5,8-Dimethoxychromone-2-carboxylic acid (VI) Et-ester (VII), obtained in 90% yield by heating 29.6 g. V in 150 ml. glacial HOAc with 8 ml. concentrated HCl, m. 173-4° (from alc.). VI, obtained in 70% yield by heating 27.8 g. VII 6 hrs. in 150 ml. glacial HOAc with 200 ml. 4N H2SO4, m. 230-1° (from HOAc), forms no oxime. Bu ester of VI obtained in 60% yield from 2.5 g. VI, 250 ml. BuOH, and 20 g. concentrated H2SO4 refluxed 6 hrs., diluted with HOH, and neutralized with NaHCO3, m. 95-6° (from 60% MeOH), forms no oxime. 6,7-Dimethoxychromone-2-carboxylic acid (C.A. 44, 7317a) (25 g.), in 400 ml. BuOH

and 140 g. concentrated H2SO4 refluxed 8 hrs., diluted with HOH, and neutralized with NaHCO3, gives 18.5 g. Bu ester, m. 131-2.5° (from 75% alc.). The presence of the MeO groups enhances the pharmacol. activity of the chromone derivs. similar to IV, but has little effect if a carboxyl group is already present; the position of the MeO groups seems to be unimportant.

IT 854846-47-2P, Chromone, 5,8-dimethoxy-2-methyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 854846-47-2 CAPLUS

CN Chromone, 5,8-dimethoxy-2-methyl-, oxime (5CI) (CA INDEX NAME)



L9 ANSWER 60 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1950:49331 CAPLUS Full-text

DOCUMENT NUMBER: 44:49331

ORIGINAL REFERENCE NO.: 44:9441h-i,9442a-d

TITLE: The structure of ginkgetin, a flavone derivative from the leaves of ginkgo trees

AUTHOR(S): Nakazawa, Koichi

CORPORATE SOURCE: Gifu Coll. Pharmacy

SOURCE: Yakugaku Zasshi (1941), 61, 228-9
From: Complete Abstracts Japan. Chem. Lit. 15, 883-5(1941).
CODEN: YKKZAJ; ISSN: 0031-6903

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

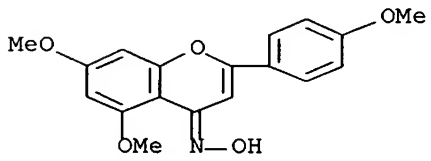
AB An alc. extract of the fallen leaves taken up with ether, extracted with 10% K2CO3, the precipitated K compound decomposed with acid, and the product recrystd. from Me2CO gives ginkgetin (I). The Me2CO-insol. portion also gives free I; yield, 0.02-0.03%. Recrystn. of I from Me2O gives yellow-white needles, m. 297°, giving a brown-purple color with alc.-FeCl3, orange-red with Mg-concentrated HCl, yellow with concentrated H2SO4, insol. in alkali bicarbonates, soluble in alkali carbonates, but precipitating on cooling. I has 2 MeO, 4 OH, and 2 CO groups (does not form an oxime). I has the composition C32H22O10 and mol. weight 572; tetraacetate, C32H18O6(OAc)4, m. 258°, gives no color with FeCl3; di-Me ether, C32H20O8(OMe)2, m. 282°, gives a brown-purple color with FeCl3, insol. in alkali (diacetate, m. 228°); tetra-Me ether, C32H18O6(OMe)4, m. 228°, gives no color with FeCl3 (oxime, m. 250°); tetra-Et ether, m. 175°, mol. weight 700°. Demethylation of I gives C30H18O10, m. 330°, whose acetate, C30H12O4(OAc)6, m. 239-40°. In general the m. ps. of I or its derivs. are 50-60° higher than those of apigenin-type compds. (acacetin, genkwanin, etc.). Fusion of I with KOH give p-HOC6H4CO2H, AcOH, phloroglucinol, and an acid, C9H10O5 (2,4,6-MeO(HO)2C6H2CH2CO2H), m. 188° (decomposition). I with alkaline KMnO4 gives no definite oxidation product but the di-Me ether gives anisic acid. Heating I with 20% aqueous KOH 3.5 hrs. gives water-soluble p-HOC6H4Ac and a water-insol. compound, C24H18O7 (II), m. 280°, easily soluble in organic solvents, gives a dark-green color with alc.-FeCl3 [semicarbazone, m. 268° (decomposition)]. The above expts. indicate that I is composed of 2 mols. of apigenin 7-Me ether (III) (genkwanin) with the suggested union binding at 3,8' or 3,6'. For comparison

acacetin di-Me ether, C16H10O3(OMe)2, m. 156-7°, was prepared from acacetin, Me2SO4, and KOH; oxime, C18H17O5N, m. 140°; di-Et ether, m. 194°. Methylation of II with MeI and K2CO3 gives a di-Me ether, C23H13O4(OMe)3, m. 224°.

IT 872302-08-4P, Flavone, 4',5,7-trimethoxy-, oxime
RL: PREP (Preparation)
(preparation of)

RN 872302-08-4 CAPLUS

CN Flavone, 4',5,7-trimethoxy-, oxime (5CI) (CA INDEX NAME)



L9 ANSWER 61 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1938:41824 CAPLUS Full-text

DOCUMENT NUMBER: 32:41824

ORIGINAL REFERENCE NO.: 32:5833g-i,5834a

TITLE: The synthesis of 5-hydroxy-6-aminoflavone

AUTHOR(S): Sugasawa, S.

SOURCE: Yakugaku Zasshi (1936), 56, 105-7
From: Chem. Zentr. 1936, II, 3669
CODEN: YKKZAJ; ISSN: 0031-6903

DOCUMENT TYPE: Journal

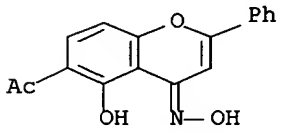
LANGUAGE: Unavailable

AB cf. C. A. 28, 6717.7; 29, 787.8. In some earlier expts. on the synthesis of primetin (cf. C. A. 28, 1345.5; 29, 160.9) work was done with 5-hydroxy-6-acetylflavone (cf. Baker, C. A. 29, 1422.4). Its oxime was caused to undergo the Beckman rearrangement to give 5-hydroxy-6- aminoflavone (I). Attempts to replace the NH2 by OH are as yet unsuccessful. 5-Hydroxy-6-acetylflavone oxime, C17H13O4N, gives yellow needles from glacial HOAc, m. 237-8°. When 1 g. of this compound is introduced into 10 cc. well cooled POCl3, warmed 3-5 min. at 70-80° and poured onto ice, yellowish brown hair-like crystals of 5-hydroxy-6-acetaminoflavone, C17H13O4N, m. 234-5° (from 1:1 alc.-glacial HOAc) are obtained. I is obtained by boiling this product 1.5-2 h. with about 20% HCl and decomposing the precipitate (probably the HCl salt) with aqueous Na2CO3; crystals from alc., m. 177°. I is golden yellow, its sulfate and HCl salt are white and difficultly soluble in water.

IT 854244-53-4P, Flavone, 6-acetyl-5-hydroxy-, oxime
RL: PREP (Preparation)
(preparation of)

RN 854244-53-4 CAPLUS

CN Flavone, 6-acetyl-5-hydroxy-, oxime (4CI) (CA INDEX NAME)



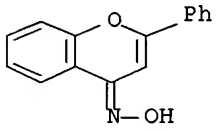
L9 ANSWER 62 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1936:61866 CAPLUS Full-text
DOCUMENT NUMBER: 30:61866
ORIGINAL REFERENCE NO.: 30:8214c-d
TITLE: A new method of oximation
AUTHOR(S): Gulati, K. C.; Ray, J. N.
SOURCE: Current Science (1936), 5, 75
CODEN: CUSCAM; ISSN: 0011-3891
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB cf. C. A. 29, 163.5. The oximes of flavone and α-naphthaflavone were obtained by reaction with NH2OH in aqueous pyridine as follows: reflux 0.1 g. 4 hrs. with 0.15 g. NH2OH.HCl in 0.5 cc. H2O and 1 cc. pyridine, and pour into dilute AcOH when cold. Crystallized from hot dilute acetone, flavone gave colorless needles, m. 237°, and α-naphthaflavone colorless needles, m. 181°.

IT 22115-89-5P, Flavone, oxime
RL: PREP (Preparation)
(preparation of)

RN 22115-89-5 CAPLUS

CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 63 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1936:22405 CAPLUS Full-text
DOCUMENT NUMBER: 30:22405
ORIGINAL REFERENCE NO.: 30:2946g-i,2947a-b
TITLE: Hydroxycarbonyl compounds. X. Coumarins and chromones from m-4-xylenol
AUTHOR(S): Flynn, Daniel G.; Robertson, Alexander
SOURCE: Journal of the Chemical Society (1936) 215-17
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

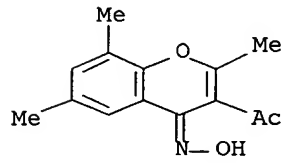
AB cf. C. A. 28, 4419.1. -m-4-Xylenol (I) and AcCH2CO2Et with 86% H2SO4 yield 4,6,8-trimethylcoumarin, m. 114-14.5°; this yields β-3,5-trimethylcinnamic acid, m. 139°, oxidation of which yields 3,5,2-Me2(MeO)C6H2Ac, whose semicarbazone m. 193°. 3,4,6,8-Tetramethylcoumarin yields 2-methoxy-α,β,3,5-tetramethylcinnamic acid, m. 139.5-40°. 4,6,8-Trimethyl-3- ethylcoumarin, m. 112.5-13°; 2-methoxy-β,3,5-trimethyl-α- ethylcinnamic acid, m. 112°. I and BzCH2CO2Et with 86% H2SO4 give 4-phenyl-6,8-di-methylcoumarin, m. 111°; PhCH2CH2CO2Et gives 3-benzyl-4,6,8-trimethylcoumarin, m. 112-13°. 3,5,2-Me2(HO) C6H2Ac, AcOEt and Na, heated 5.5 hrs. on the water bath, give 2-hydroxy-β-acetyl-3,5-dimethylacetophenone, m. 85°; AcOH-HCl yields 2,6,8-trimethylchromone, m. 125°; this was also prepared from I, AcCH2CO2Et and P2O5 on heating 3 hrs.; condensation with piperonal gives 2-(3',4'-methylenedioxystyryl)-6,8-dimethylchromone, pale yellow, m. 195°. The propionate of I, b20 124-5°, and AlCl3, heated at 130-40° for 5 hrs., give 2-hydroxy-3,5-dimethylpropiophenone, m. 52-3° (deep blue FeCl3 reaction); Ac2O and AcONa give 2,3,6,8-tetramethylchromone, m. 136-7°; this also results from

I and AcCHMeCO2Et with P2O5: the 2-(3',4'-methylenedioxystyryl) derivative, yellow, m. 196°. The butyrate of I b17.5 132-3°; 2-hydroxy-3,5-dimelhylbutyrophenone, b30 145-50°, m. 30° (84% yield) (deep blue FeCl3 reaction); acetylation gives 2,6,8-trimethyl-3-ethylchromone, m. 112.5°, which also results from I and the requisite ester; the 2-(3',4'-methylenedioxystyryl) derivative, pale yellow, m. 202-3°. 3,5,2-Me2(HO)C6H2Ac (II), Ac2O and AcONa give 2,6,8-trimethylchromone, m. 125°, and the 3-Ac derivative (oxime, m. 119°). Refluxing II and EtCO2Et with Na for 3 hrs. gives 6,8-dimethyl-2-ethylchromone, m. 109-10°. Heating II with (EtCO)2O and EtCO2Na at 210° for 10.5 hrs. gives 3,4,6,8-tetramethylcoumarin and 3-propionyl-6,8-dimethyl-2-ethylchromone (oxime, m. 93°). Heating II with Bz2O and BzONa gives 3-benzoyl-6,8-dimethylflavone, m. 191-2°.

IT 859805-98-4P, Chromone, 3-acetyl-2,6,8-trimethyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 859805-98-4 CAPLUS

CN Chromone, 3-acetyl-2,6,8-trimethyl-, oxime (3CI) (CA INDEX NAME)



L9 ANSWER 64 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1926:11317 CAPLUS Full-text

DOCUMENT NUMBER: 20:11317

ORIGINAL REFERENCE NO.: 20:1411g-i,1412a-h

TITLE: Action of hydroxylamine on chromones

AUTHOR(S): Wittig, Georg; Bangert, Fritz

SOURCE: Berichte der Deutschen Chemischen Gesellschaft [Abteilung] B: Abhandlungen (1925), 58B, 2636-42
CODEN: BDCBAD; ISSN: 0365-9488

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

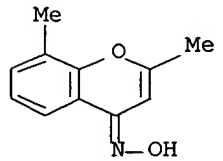
AB Although the C : O group of chromones reacts with extraordinary sluggishness with ketone reagents it shows a striking reactivity with NH2OH. From the observations of Harries on analogous compds. (Ann. 330, 190(1904)) it might be expected that NH2OH first adds at the double bond of the pyrone ring, with formation of a hydroxyaminochromanone (I) whose C:O group can now be oximated with ease, acidification of the product (II) splitting off NH2OH and yielding the chromone oxime (III). As a matter of fact, NH2OH in neutral solution with 2,8-dimethylchromone (IV) and 2,8-dimethyl-4-thiochromone gives the oxime (V) of IV. On the other hand, if IV is oximated in alkaline solution and the still warm reaction mixture is acidified with a dilute mineral acid, V is again obtained but if AcOH is cautiously added instead of the mineral acid there seps. a compound C11H14O8N2 (VI, R = 3,2-Me(HO)C6H2) which is also obtained from 6,2-Me(AcCH2CO)C6H3OH (VII) with NH2OH, showing that in the oximation of IV the pyrone ring is ruptured, with formation of the dioxime VI from which hot mineral acids hydrolyze the oxime group furthest from the C6H6 ring with formation of V. With cold acids VI gives, together with V, an alkali-soluble isomer (VIII). V and VIII cannot be converted into each other by concentrated alkalies or acids, and VIII, which, unlike V, gives a cornflower-blue color with FeCl3, can also be obtained from VI by the action

of alc. NH3, i. e., conditions under which chromone formation is impossible. VIII can therefore be only the hydroxyphenylisoxazole IX or X. It can also be obtained by heating VI at 160°. Since VI in general shows a tendency to split off the oxime group furthest from the C6H6 ring, IX is probably the correct formula for VIII. Alkaline oximation of 2,6-dimethylchromone (XI) yields an extremely unstable compound, apparently a hydroxyamino oxime (XII) in which the NHOH group is situated away from the C6H6 ring, as with cold acids it readily yields the oxime (XIII) of XI, also obtained with hot mineral acids without the formation of the hydroxyphenylisoxazole (XIV) (X, R = 5,2-Me(HO)C6H3), which can be obtained, together with XIII, by fusing the dioxime (XV) of 4,2-Me(AcCH2CO)C6H3OH (XVI). With alc. NH3, XV unexpectedly gave XII; apparently, in the absolute alc. NH3 added on the oxime group furthest from the C6H6 ring and on acidification was replaced by H2O; an analogous NH3 addition compound is probably an intermediate product in the formation of VIII from VI. On long heating with NaOH, XV gives an alkali-soluble compound C11H12O2N2 which by the Schotten-Baumann method yields a dibenzoate; apparently XV first forms the anhydride (cf. 2,6-dimethyl-3-acetochromone dioxime, preceding abstract) in which, under the further action of the alkali there occurs a shifting of the double bond with formation of the isoxdiazine XVII or XVIII (R = 5,2-Me(HO)C6H3); at the same time is formed an isomeric isoxdiazine which with Ac2O and NaOAc gives a diacetate and which doubtless also has 1 of the structures XVII or XVIII; the solubility in Na2CO3 of the 1st isomer and the slight solubility of the latter in NaOH indicate that they are XVIII and XVII, resp. 2-Acetylaceto-6-methylphenol dioxime (VI), obtained in 5.5 g. yield from 5 g. VII or in 75% yield from IV, m. 148-9° (slight decomposition), unchanged by heating with H2O under pressure. V, obtained almost quant. from VI in boiling aqueous alc. HCl, in 35% yield from the thiochromone with NH2OH in aqueous alc. and in about 75% yield from IV, m. 145.5-6.0°. α-[2-Hydroxy-3- methylphenyl]-γ-methylisoxazole (VIII), obtained in 80% yield from VI at 150-60°, in 30% yield from VI and alc. NH8, and in 0.3 g. yield, together with 0.4 g. V, from 1 g. VI in MeOH with cold 0.5 N HCl, m. 90.5-1.0°. 2-[α-Hydroximino-γ-hydroxamino-γ- hydroxyacetylaceto]-4-methylphenol (XII), m. 70-3°, loses H2O and solidifies and then has the m. p., 122-2.5°, of 2-acetylaceto-4-methylphenol dioxime (XV); both XII and XV give a blue color with alc. FeCl3. XIII, m. 184-5°. α-[2-Hydroxy-5- methylphenyl]-γ-methylisoxazole (XIV) (yield, 60%), m. 53-4°. 5-[2'-Hydroxy-5'-methylphenyl]3-methyl-1,2,6-isoxdiazine (XVIII) (3 g. from 4 g. XV refluxed 6-7 hrs. in excess of 2 N NaOH), m. 168-9° (slight decomposition), gives an olive-green color with FeCl3; dibenzoate m. 123.5-4.0°. 3,5-Isomer (XVIII) (yield, 0.4 g.), m. 185-7° (slight decomposition), gives no color with FeCl2, soluble in hot acids and alkalies and seps. unchanged on cooling; diacelate, m. 155.5-6.0°.

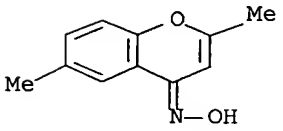
IT 56686-36-3P, Chromone, 2,8-dimethyl-, oxime 56686-37-4P, Chromone, 2,6-dimethyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 56686-36-3 CAPLUS

CN 4H-1-Benzopyran-4-one, 2,8-dimethyl-, oxime (9CI) (CA INDEX NAME)



RN 56686-37-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 2,6-dimethyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 65 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1920:19461 CAPLUS Full-text
DOCUMENT NUMBER: 14:19461
ORIGINAL REFERENCE NO.: 14:3633c-i,3634a-d
TITLE: Ring formation. I. Unsaturated ketones and chromanones from p-cresol
AUTHOR(S): v. Auwers, Karl; Lammerhirt, Elisabeth
CORPORATE SOURCE: Univ. Marburg
SOURCE: Ann. (1920), 421, 1-58
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 14:19461

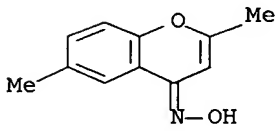
AB 3,6-Dimethylchromanone (C. A. 11, 2793) has been synthesized from p-MeC6H4OMe and Me2CBrCO2Cl in CS2 by the action of AlCl3, β-chloroisobutyro-p-cresol, yellow oil, b13 135-7°, being an intermediate product. 3-Bromo-3,6-dimethylchromanone, glistening prisms, m. 70-1°. Heated with PhNMe2 for 0.5 hr., this yields 3,6-dimethylchromone, glistening needles, m. 61-2°, b760 299-301°, b15, 165-8°. The structure of the chromone was established by synthesis from MeC6H3(OH)COEt and (CO2Et)2 by means of Na, and then heating with concentrated HCl, the 1st product being 3,6-dimethylchromone-2-carboxylic acid, needles, m. 234-6°; heated over the free flame, CO2 is evolved and the chromone formed. The reverse reaction is brought about by heating the chromone in EtONa for 0.5 hr. o-Propio-p-cresol p-nitrophenylhydrazone, orange-red, compact needles, m. 188-9°. 6-Methylchromone, from the Br deriv (C. A. 9, 84) by the action of PhNMe2, small, flat, glistening prisms, m. 88-9°. This was synthesized from HOC6H3MeCOMe and (CO2Et)2 by the action of Na, the intermediate product being ethyl 5-methyl-2-hydroxybenzoylpyroracemate, glistening, flat needles, m. 78-9°, which, upon hydrolysis, yielded 6-methylchromone-2-carboxylic acid, and this in turn the desired chromone. 2,6-Dimethylchromanone results by the action of MeC6H4OMe and MeCH:CHCOCl with 2 mols. AlCl3; glistening prisms, m. 54-5°, b10-5 138°, b20 152-3°, b26 162-3°, b760 262-3°. Semicarbazone, prisms, m. 203°. A by-product, separated by shaking the Et2O solution with NaOH, is 3,4-dimethyl-7-hydroxyhydrindone which seps. as the Na salt. By the use of 1 mol. AlCl3 there also results o-propenyl-p-cresyl ketone (o-crotonyl-p-cresol), golden transparent prisms and plates, m. 65-6°, b. 277-8°. MeCHClCH2CO2H, b20 110-3°, d420.2 1.1861, d419.85 1.1865, n 1.43992, 1.44213, 1.44828, 1.45327 for α, D, β and γ, at 19.85°, nD20 1.4421. β-Chlorobutyryl chloride, b21 51-3°, d420.06 1.2165, d420 1.217, n 1.44833, 1.45085, 1.45774, 1.46341 for α, D, β and γ at 20.05°, nD20 1.4511; gives with p-MeC6H4OMe β-chlorobutyro-p- cresol, b20 167-70°. Heated with dilute Na2CO3, this gives 2,6-dimethylchromanone. 3-Bromo-2,6-dimethylchromanone, glistening prisms, m. 104-5°. Heated with PhNMe2, this gives 2,6-dimethylchromone, compact needles, m. 102-3°. 5-Methyl-2-methoxybenzoylacetone, from MeOC6H3MeCOMe and AcOEt, yellow oil, b15 182-3°, d417.9 1.1196, d420 1.11, n 1.57582, 1.58562, 1.61562 for α, D and β at 17.9°, nD20 1.5847. Heated with HI, this gives 2,6-dimethylchromanone. p-Cresyl

crotonate, b27 153-5°, d420 1.059, nD20 1.5138, when treated with AlCl3 at 120° gave only 3,4-dimethyl-7-hydroxyhydrindone. The interaction of 1 mol. each of p-MeC6H4OMe, Me2C:CHCOCl and AlCl3 gives 2,2,6-trimethylchromanone, isolated as the semicarbazone, fine needles, m. 199-200°, and isobutenyl cresyl ketone, whose semicarbazone, m. 148-9°. With 2 mols. AlCl3 there results o-isobutenyl p-cresyl ketone, S-yellow prisms and long needles, m. 50°, b15 150-60°, d45.38 1.0376, nα 1.56280, nD 1.57178 With dilute NaOH it gives 2,2,6- trimethylchromanone; MeONa, boiling alc. HCl or H2SO4 or simple distillation causes the same change. It may be reduced to o-isovalero-p-cresol. Saturating a solution in AcOH with HCl gives o-[β-chloroisovalero]-p-cresol, compact prisms, m. 53-55°. o-[α,β-Dibromoisovalero]-p- cresol, pale yellow needles, m. 70-1°. A small amount of 3,3,4-trimethyl-7-hydroxyhydrindone, m. 67-8°, is also obtained in the above reaction, small compact prisms, m. 67-8°; semicarbazone, fine needles, m. 201-2°. 3,3-Dibromo-6-methylchromanone, compact, glistening prisms, m. 119-20°. Oxamino-oxime of 6-methylchromone, by the action of dilute alc. upon the chromone, prisms from MeOH, m. 143-4°. 3,6-Dimethylchromanone oxime, flat needles and prisms, m. 129-30°. p-Nitrophenylhydrazone, orange-red crystals, m. 179°. 3,6-Dimethylchromone oxime, prisms, m. 131-2°. 3,4,6-Trimethyl-4-chromanol, by the action of MeMgI upon dimethylchromanone, compact, flat prisms, m. 124°. 3,4,6-Trimethyl-α-chromene, prepared by the dehydration of the above alc. with P2O5, b18 135-6°. 2,6-Dimethylchromanone oxime, compact, glistening needles, m. 135°. Phenylhydrazone, compact prisms, m. 133°. p-Nitrophenylhydrazone, orange-red needles, m. 229-30°. 3,3-Dibromo-2,6-dimethylchromanone, compact, compact prisms, m. 100-1°. 2,6-Dimethylchromone oxime, fine needles, m. 151-2°. The Na salt is easily soluble in dilute NaOH. 2,4,6-Trimethyl-4-chromanol, small prisms, m. 89-90.5°. 2,4,6-Trimethyl-α-chromene, b25 138.5-9.5°. 2,2,6-Trimethylchromanone oxime, compact, glistening crystals, m. 130-1°. p-Nitrophenylhydrazone, orange-red glistening needles, m. 202°. 3-Bromo derivative. 3,3-Dibromo derivative, needles, m. 85°.

IT 56686-37-4P, Chromone, 2,6-dimethyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 56686-37-4 CAPLUS

CN 4H-1-Benzopyran-4-one, 2,6-dimethyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 66 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1908:7441 CAPLUS Full-text

DOCUMENT NUMBER: 2:7441

ORIGINAL REFERENCE NO.: 2:1709g-i,1710a-b

TITLE: Two Monohydroxy-α-Naphthoflavonols

AUTHOR(S): v. Kostanecki, St.

CORPORATE SOURCE: Univ. Lab., Bern

SOURCE: Berichte der Deutschen Chemischen Gesellschaft (1908), 41, 783-6
CODEN: BDCGAS; ISSN: 0365-9496

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

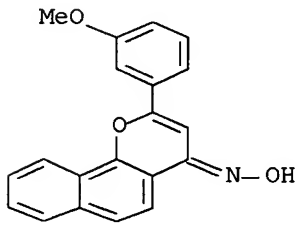
GI For diagram(s), see printed CA Issue.

AB 4'-Methoxy-α-naphthoflavanone (I below) from 2-anisalaceto-1- naphthol, HCl and MeOH. Colorless needles, m. 148°. Isonitroso derivative, yellow needles, m. and decomposes 169-70°. It gives orange colors with cobalt mordants and yellow ones with mordants of uranium, cadmium and lead. 4'-Methoxy-α-naphthoflavonol, by hydrolysis of the preceding compound with AcOH and dilute H2SO4. Slender, yellow needles, m. 249°. It gives light yellow colors with aluminum mordants and a light green, intensely fluorescent solution with concentrate H2SO4. Sodium salt, yellow and sparingly soluble. Acetyl derivative, colorless interlaced needles, m. 196°. 4'-Hydroxy-α-naphthoflavonol (II), from the methoxy compound and HI. Pale yellow plates, m. 293°. It gives light yellow colors with aluminum mordants. Concentrate H2SO4 dissolves it with a pale yellow color and an intense light green fluorescence; in aqueous NaOH the color is yellow with a greenish fluorescence. Diacetyl derivative, colorless needles, m. 181°. 3'-Methoxy-2-benzalaceto-1-naphthol, HOC10H6COCH:CHC6H4OMe, from m-methoxybenzaldehyde and 2-aceto-1-naphthol; orange-red needles, m. 115°. With concentrate H2SO4 the crystals darken and give a red solution. 3'-Methoxy-α-naphthoflavanone, from the preceding compound and HCl. Colorless needles, m. 130°. Isonitroso derivative, yellow crystalline powder, m. and decomposes 151°. It gives a pale yellow solution with dilute aqueous NaOH and orange colors with cobalt mordants. 3'-Methoxy-α-naphthoflavonol, yellow needles, m. 185°. In concentrate H2SO4, its solution is light yellow. With aluminum mordants it dyes pale yellow. Sodium salt, yellow and sparingly soluble. Acetyl derivative, colorless needles, m. 165°. 3'-Hydroxy-α-naphthoflavonol, from the methoxy derivative and HI. Lustrous pale yellow prismatic needles with 1EtOH, m. 248°. It dyes pale yellow with aluminum mordants. In concentrate H2SO4 the solution is pale yellow with a feeble greenish fluorescence. Sodium salt, slender yellow needles. In highly dilute solution it has a feeble greenish fluorescence.

IT 861550-15-4P, 7,8-Benzoflavanone, 3'-methoxy-, oxime
RL: PREP (Preparation)
(preparation of)

RN 861550-15-4 CAPLUS

CN 7,8-Benzoflavanone, 3'-methoxy-, oxime (1CI) (CA INDEX NAME)



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FULL ESTIMATED COST	366.20	585.91

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL

	ENTRY	SESSION
CA SUBSCRIBER PRICE	-51.48	-51.48

SESSION WILL BE HELD FOR 120 MINUTES
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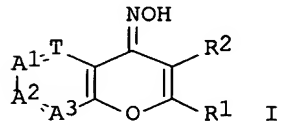
L6 ANSWER 1 OF 7 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 2004:825133 CAPLUS Full-text
DOCUMENT NUMBER: 141:332051
TITLE: Preparation of substituted chromen-4-one oximes as inhibitors of protein kinases
INVENTOR(S): Green, Jeremy; Aronov, Alex; Pierce, Albert C.
PATENT ASSIGNEE(S): USA
SOURCE: U.S. Pat. Appl. Publ., 47 pp.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004198750	A1	20041007	US 2004-808678	20040325
AU 2004230841	A1	20041028	AU 2004-230841	20040325
CA 2522595	A1	20041028	CA 2004-2522595	20040325
WO 2004092154	A1	20041028	WO 2004-US9145	20040325
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1615906	A1	20060118	EP 2004-758959	20040325
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, PL, SK				
JP 2006522124	T	20060928	JP 2006-509283	20040325

PRIORITY APPLN. INFO.: US 2003-460042P P 20030403
WO 2004-US9145 W 20040325

OTHER SOURCE(S): MARPAT 141:332051
GI



I

AB The title compds. [I; R1 = LmR, LmAr1, LmCy1; L = S, O, NR, alkylidene wherein up to two non-adjacent methylene units of L are optionally replaced by S, O, CO, etc.; m = 0-1; Ar1 = (un)substituted 5-7 membered monocyclic or 8-10 membered bicyclic ring having 0-5 heteroatoms; Cy1 = (un)substituted 3-7 membered (un)saturated monocyclic ring having 0-3 heteroatoms or 8-10 membered (un)saturated bicyclic ring having 0-5 heteroatoms; R = H, alkyl; R2 = H, CN, SR, OR, etc.; T = N, CR3; A1-A3 = N, CR4; provided that no more than two of T, A1-A3 are N atom; R3 = H, halo, NO2, etc.; R4 = halo, NO2, CN, etc.; with provisos], useful as inhibitors of protein kinases, were prepared E.g., a 2-step synthesis of 2-(4-methoxyphenyl)-8-methylchromen-4-one oxime, starting from 8-methyl-4'-methoxyflavone, was given. The exemplified compds. I were

tested and found to inhibit CDK-2, cMET, GSK-3, SYK, ZAP-70, FLT-3, JAK-3, p70S6K, TAK-1, and IRAK-4. The invention also provides pharmaceutically acceptable compns. comprising said compds. I and methods of using the compns. in the treatment of various disease, conditions, or disorders.

IT 59835-92-6P 115663-23-5P 140885-79-6P
304691-31-4P 321976-78-7P 769948-78-9P
769948-79-0P 769948-80-3P 769948-81-4P
769948-82-5P 769948-83-6P 769948-84-7P
769948-85-8P 769948-86-9P 769948-87-0P
769948-88-1P 769948-89-2P 769948-90-5P
769948-91-6P 769948-92-7P 769948-93-8P
769948-94-9P 769948-95-0P 769948-96-1P
769948-97-2P 769948-98-3P 769948-99-4P
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769949-03-3P 769949-04-4P 769949-06-6P
769949-07-7P 769949-08-8P 769949-09-9P
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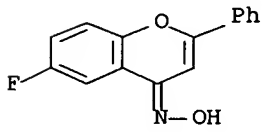
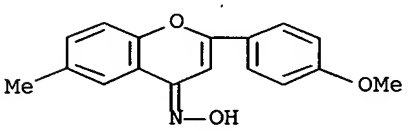
769949-16-8P 769949-17-9P
RL: PAC (Pharmacological activity); SPN (Synthetic preparation); THU (Therapeutic use); BIOL (Biological study); PREP (Preparation); USES (Uses)
(preparation of substituted chromen-4-one oximes as inhibitors of protein kinases)

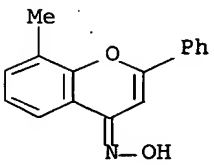
RN 59835-92-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-phenyl-, oxime (CA INDEX NAME)



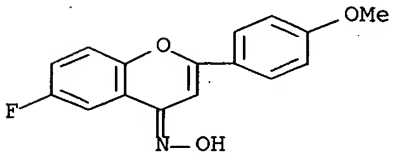
RN 115663-23-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-6-methyl-, oxime (CA INDEX NAME)



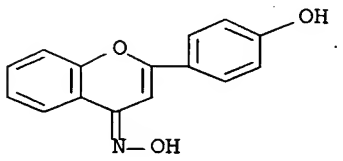
RN 140885-79-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 8-methyl-2-phenyl-, oxime (CA INDEX NAME)



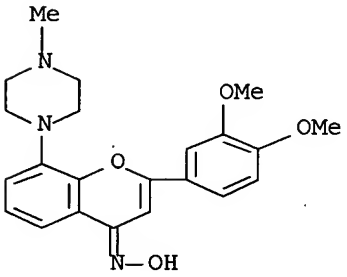
RN 304691-31-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-(4-methoxyphenyl)-, oxime (CA INDEX NAME)



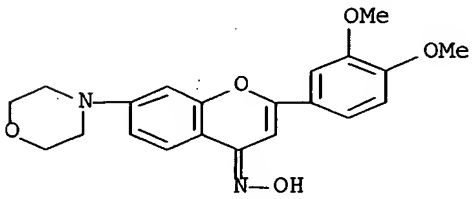
RN 321976-78-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-, oxime (CA INDEX NAME)



RN 769948-78-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-8-(4-methyl-1-piperazinyl)-, oxime (CA INDEX NAME)

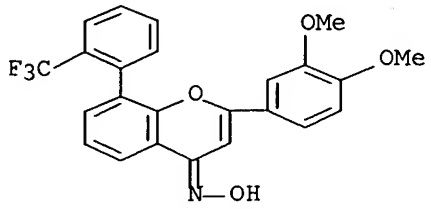


RN 769948-79-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-7-(4-morpholinyl)-, oxime (CA INDEX NAME)

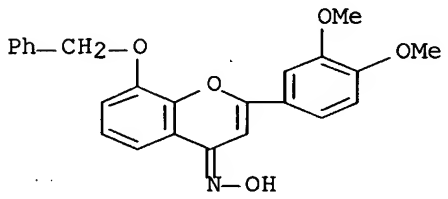


RN 769948-80-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-8-[2-

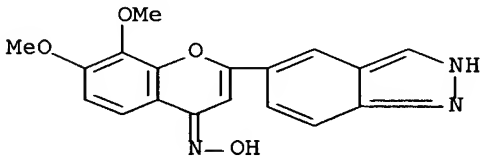
(trifluoromethyl)phenyl]-, oxime (CA INDEX NAME)



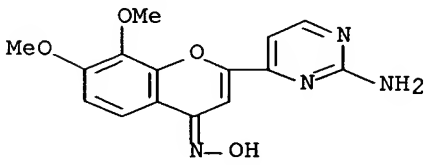
RN 769948-81-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-8-(phenylmethoxy)-, oxime (CA INDEX NAME)



RN 769948-82-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2H-indazol-5-yl)-7,8-dimethoxy-, oxime (CA INDEX NAME)



RN 769948-83-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2-amino-4-pyrimidinyl)-7,8-dimethoxy-, oxime (CA INDEX NAME)



RN 769948-84-7 CAPLUS
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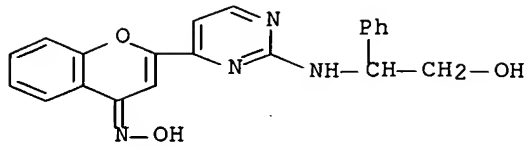
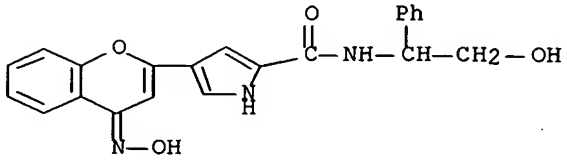
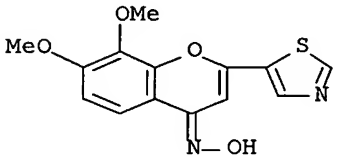


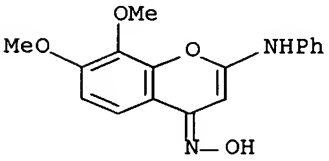
RN 769948-85-8 CAPLUS
CN 1H-Pyrrole-2-carboxamide, 4-[4-(hydroxyimino)-4H-1-benzopyran-2-yl]-N-(2-hydroxy-1-phenylethyl)- (CA INDEX NAME)



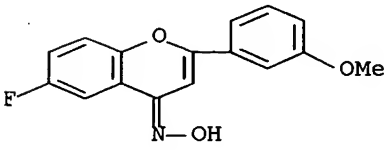
RN 769948-86-9 CAPLUS
CN 4H-1-Benzopyran-4-one, 7;8-dimethoxy-2-(5-thiazolyl)-, oxime (CA INDEX NAME)



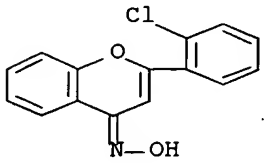
RN 769948-87-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 7,8-dimethoxy-2-(phenylamino)-, oxime (CA INDEX NAME)



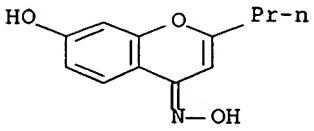
RN 769948-88-1 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-(3-methoxyphenyl)-, oxime (CA INDEX NAME)



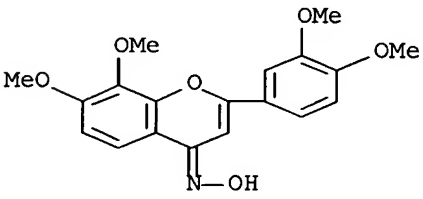
RN 769948-89-2 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(2-chlorophenyl)-, oxime (CA INDEX NAME)



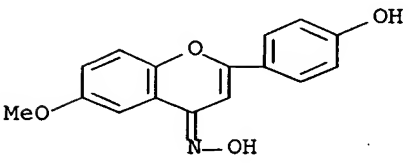
RN 769948-90-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-hydroxy-2-propyl-, oxime (CA INDEX NAME)



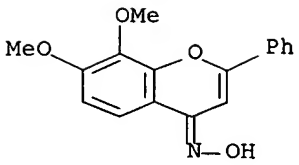
RN 769948-91-6 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-7,8-dimethoxy-, oxime (CA INDEX NAME)



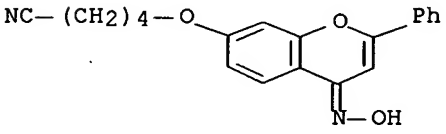
RN 769948-92-7 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-6-methoxy-, oxime (CA INDEX NAME)



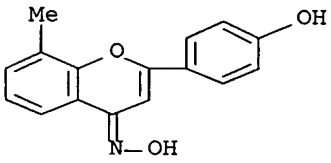
RN 769948-93-8 CAPLUS
CN 4H-1-Benzopyran-4-one, 7,8-dimethoxy-2-phenyl-, oxime (CA INDEX NAME)



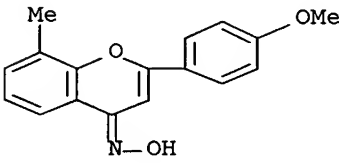
RN 769948-94-9 CAPLUS
CN Pentanenitrile, 5-[[4-(hydroxyimino)-2-phenyl-4H-1-benzopyran-7-yl]oxy]- (CA INDEX NAME)



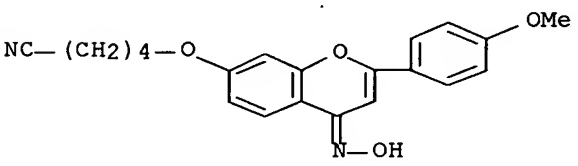
RN 769948-95-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-8-methyl-, oxime (CA INDEX NAME)



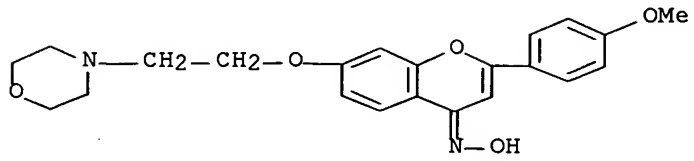
RN 769948-96-1 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-8-methyl-, oxime (CA INDEX NAME)



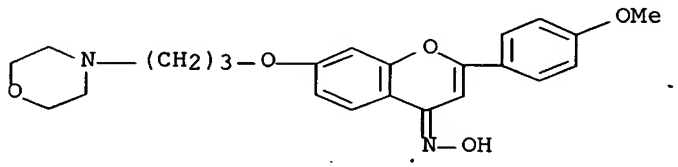
RN 769948-97-2 CAPLUS
CN Pentanenitrile, 5-[[4-(hydroxyimino)-2-(4-methoxyphenyl)-4H-1-benzopyran-7-yl]oxy]- (CA INDEX NAME)



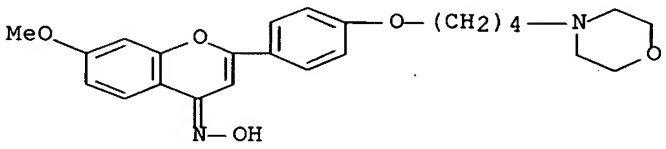
RN 769948-98-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[2-(4-morpholinyl)ethoxy]-, oxime (CA INDEX NAME)



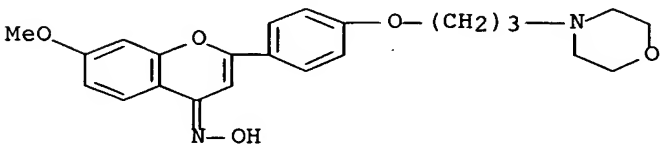
RN 769948-99-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[3-(4-morpholinyl)propoxy]-, oxime (CA INDEX NAME)



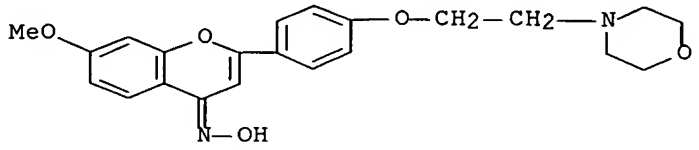
RN 769949-00-0 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[4-(4-morpholinyl)butoxy]phenyl]-, oxime (CA INDEX NAME)



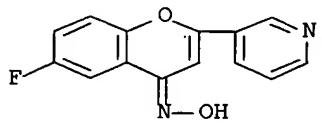
RN 769949-01-1 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[3-(4-morpholinyl)propoxy]phenyl]-, oxime (CA INDEX NAME)



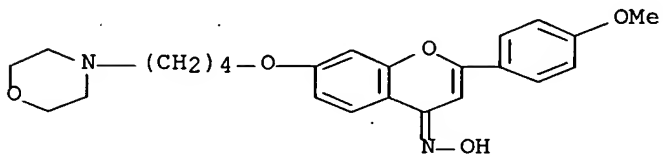
RN 769949-02-2 CAPLUS
CN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[2-(4-morpholinyl)ethoxy]phenyl]-, oxime (CA INDEX NAME)



RN 769949-03-3 CAPLUS
CN 4H-1-Benzopyran-4-one, 6-fluoro-2-(3-pyridinyl)-, oxime (CA INDEX NAME)



RN 769949-04-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[4-(4-morpholinyl)butoxy]-, oxime (CA INDEX NAME)



RN 769949-06-6 CAPLUS
CN Acetonitrile, [[4-(hydroxyimino)-7-methoxy-2-phenyl-4H-1-benzopyran-8-yl]oxy]- (9CI) (CA INDEX NAME)